Raleigh/Wake City-County
Bureau of Identification
Crime Laboratory Division

Drug Chemistry Unit
DRUG CHEMISTRY TECHNICAL PROCEDURES

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1: Technical Procedures for Drug Chemistry Analysis

1. **Purpose/Scope -** This procedure provides direction for analysis for controlled substances in the Raleigh/Wake City-County Bureau of Identification. It includes the analysis schemes, sampling procedures, minimum requirements for identification and reporting of controlled substances, as specified in the North Carolina Controlled Substances Act, and federally controlled substances as specified in 21 CFR § 1308 and 21 USC §801 et seq.

2. **Definitions**

   2.1. Sample Selection – A practice of selecting items to test, or portions of items to test, based on training, experience and competence. In sample selection, there is no assumption about homogeneity.

   2.2. Sampling Procedure – A defined procedure used to collect a sample or samples from the larger whole, to ensure that the value obtained in the analysis is representative of the whole. The sampling procedure may include details about size and number of sample(s) to be collected, locations from which to collect the sample(s), and a method to ensure the homogeneity of the larger whole (or to make it so.)

   2.3. Sampling – Taking a part of a substance, material or product for testing in order to reach a conclusion, make an inference about, and report on the whole. Sampling should only be used when there is a reasonable assumption of homogeneity of the whole.

   2.4. Sampling Plan – For an item that consists of a multi-unit population (e.g., tablets, baggies, bindles), a sampling plan is a statistically valid approach to determine the number of sub-items that must be tested in order to make an inference about the whole population.

   2.5. Administrative Sample Selection - A sample selection method used for all multi-unit items containing pharmaceutical preparations without indications of tampering. This sample selection method is also used for multi-unit non-pharmaceutical items when a threshold does not apply. No inferences about unanalyzed material are made.

   2.6. Threshold Sample Selection - A sample selection method used when the material, dosage units or tablets present in a submission meet a threshold and the individual analysis of the packages, units or tablets is practicable. The practicability of analysis is determined by the analyzing Drug Chemist based on their training and experience. No inferences about unanalyzed material are made.

   2.7. Hypergeometric Sampling Plan - A statistically-based sampling plan that allows analysis of a portion of a population and a statistical inference about the whole population stating that the material was analyzed with a statistical sampling plan that demonstrates with 95 % confidence that at least 90 % of the material contains the identified controlled substance(s). The hypergeometric sampling plan is used when there are ten or more packages, units or tablets and the threshold sampling plan is not practicable. The practicability of analysis is determined by the analyzing Drug Chemist based on their training and experience.

   2.8. Population - A carefully inspected group of packages, units or tablets found to be homogenous.

3. **Abbreviations**

   3.1. Common chemical terminology and unit abbreviations may be used

3.2. approx or apx – approximately
3.3. bicarb – sodium bicarbonate
3.4. coc – cocaine
3.5. conc - concentration
3.6. c̄ – containing
3.7. d – dated
3.8. diff – difference
3.9. ext – extract / extraction
3.10. GCMS, GC-MS, or GC/MS – gas chromatography–mass spectrometry
3.11. gpm – green plant material
3.12. hs – heat sealed
3.13. hex – hexane
3.14. IR – infrared spectroscopy
3.15. i – initialed
3.16. init – initial
3.17. k – knotted
3.18. LTC – labeled to contain
3.19. lg - large
3.20. mat'l - material
3.21. me – manila envelope
3.22. med - medium
3.23. NIST – National Institute of Standards and Technology
3.24. NQCC – negative quality control check
3.25. NCS – no controlled substances
3.26. NSR – no significant reaction
3.27. ow – off-white
3.28. owhm – off-white hard material
3.29. owm – off-white material
3.30. owp – off-white powder
3.31. p – page
3.32. pg – page
3.33. pm – plant material
3.34. pb – plastic bag
3.35. pbc – plastic bag corner
3.36. pos - positive
3.37. PQCC – positive quality control check
3.38. prelim(s) – preliminary testing(s)
3.39. QCC – quality control check
3.40. rgt – reagent
3.41. rec'd – received
3.42. ref – reference
3.43. ret'd - returned
3.44. sch – schedule
3.45. s - sealed
3.46. sm - small
3.47. std(s) – standard(s)
3.48. STR – straight

3.49. sub – subtract
3.50. tp – tan powder
3.51. temp – temperature
3.52. tran(s) – transfer(s)
3.53. UV – ultraviolet spectroscopy
3.54. u – unsealed
3.55. wt – weight
3.56. we – white envelope
3.57. wp – white powder
3.58. w/ or $\overline{w}$ – with
3.59. zpb – ziploc plastic bag

4. Equipment, Materials and Reagents

4.1. Refer to the Drug Chemistry Unit Technical Procedures

5. Procedure

5.1. Laboratory facilities are subjected to environmental monitoring, refer to the Drug Chemistry Unit Technical Procedure for General Laboratory Equipment.

5.2. The analyzing Drug Chemist shall have this procedure readily available at the location of any sampling.

5.3. Use good laboratory practices at all times to maintain evidence integrity and minimize the risk of cross contamination.

5.3.1. Material from individual packages, units or tablets shall not be combined.

5.3.2. Have only one item of evidence open for analysis at a time.

5.3.3. Remove evidence to be weighed from its packaging and return it to its packaging as close as practicable to the balance in use.

5.3.4. Handle evidence carefully to minimize the risk of loss, contamination of other evidence and contamination of the work area.

5.3.4.1. Change gloves and / or wash hands between evidence items.

5.3.4.2. Ensure that the work area is clean prior to opening an evidence item and after analysis of each evidence item.

5.3.5. If any evidence is spilled or dropped, immediately check the evidence to ensure that all material / units are present.

5.3.5.1. Record the occurrence in the case file.

5.3.5.2. Clean the area thoroughly to ensure that contamination of other evidence and the work area does not occur.

5.3.6. Maintain materials used in analysis that come in direct contact with evidence in closed cabinets or drawers. These materials include but are not limited to weigh papers, spatulas, pipettes, test tubes, vials, beakers, spot plates, microscope slides, autosampler vials and caps.

5.3.7. Drug Chemists are responsible for laboratory housekeeping which ensures a clean and safe working environment.

5.3.7.1. Refer to the Crime Laboratory Health and Safety Manaual and the included Chemical Hygiene Plan.

5.3.7.2. Label all extraction vessels with identifying information such as case number / item number and dispose of the contents and the vessels properly prior to the end of each workday.

5.3.7.3. Keep the laboratory work benches, fume hoods and floors free of all items unnecessary for analysis activities at all times.

5.3.7.4. Wash used spot wells, glassware, etc. prior to the end of each working day. Dry and store items in closed drawers or cabinets immediately after washing.

5.3.7.5. Perform cleaning weekly, at a minimum, to ensure that dust and debris do not accumulate and items unnecessary for analysis are removed. The floors, fume hoods and laboratory work benches should be visibly free from dust and debris at all times.

5.3.7.5.1. Records

5.3.7.5.1.1. Record the weekly cleaning and any comments on the weekly cleaning log form.

5.4. Label analyzed individual packages, units or tablets and data generated to ensure that analysis data can be matched with the material it represents.

5.5. Record notes which will allow another Drug Chemist to repeat the analysis under conditions as close as possible to the original, evaluate the data, interpret the results, and form an independent conclusion.

5.6. Record all analyses and observations on a Drug Chemistry worksheet form as close as possible to the time performed.

5.6.1. The Drug Chemistry Additional Page Worksheet form may be used if additional space is needed.

5.6.2. The Drug Chemistry Additional Weight Worksheet form may be used when the use of the Drug Chemistry Worksheet form is impracticable to record multiple weights of material that is not analyzed.

5.6.3. Use the Drug Chemistry Hypergeometric Weight Estimation Worksheet form when Hypergeometric Sampling is used and the weight of a population is estimated.

5.6.4. The Drug Chemistry Summed Weight Worksheet form may be used when multiple weights are added for reporting.

5.7. Prior to examination, ensure that the OSSI RMS entry is accurate. Notify the Quality Manager of any discrepancies. Record the date(s) of examination as "Date started" and "Date completed" on the Drug Chemistry Cover Sheet form. The completion date is the date when all data has been incorporated into a recorded conclusion and the case file is submitted for technical review.

5.8. Record a complete description of the evidence received for each submission. Include a description of the material, number of units or dosage units (including any blotter paper perforations), all packaging, condition of seals, and submitting agency item numbers.

5.8.1. Evaluate the evidence received against the submission form. If a discrepancy occurs have another Drug Chemist evaluate the evidence. Record discrepancies on a Drug Chemistry worksheet form and have the second Drug Chemist verify the discrepancy with initials and date. If necessary, refer to the Administrative Procedure for Review of Requests, Tenders and Contracts for Laboratory Services.

5.8.1.1. If a major discrepancy occurs, e.g., a missing item of evidence, or tampering is suspected notify the Quality Manager.

5.8.1.2. When an individual with evidence in their immediate custody leaves the work area during the workday for a short time (e.g., restroom break, meal break) the evidence in the work area must be secured by locking it in a locked cabinet in the work area.

5.8.1.3. When evidence is to be left unattended for an extended period (i.e., longer than a meal break) the evidence must be secured by returning it to the unit secure area for evidence storage, room C1398.

5.8.1.4. When the work area is to be left unattended it must be secured by locking the hallway door for room C1399. Additionally, the door for room C1401 must be locked when the work area is to be left unattended.

5.8.1.5. The hallway door for room C1400 must remain locked at all times.

5.9. Evaluate submissions containing multiple items and/or multiple types of material in a single item (sub-items) to determine which items to analyze. At a minimum evaluate the submission and information received or contained in the submission, the type of charge, the location found, the

possibility of elevated charges if multiple items are analyzed and the nature of the item (biohazard, sharps hazard, amount of material present.) If necessary, record information from the submission on the Drug Chemistry worksheet.

5.9.1. Typically, do not analyze residues if another item in the submission has been found to contain a controlled substance that is the same or higher schedule than suspected in the residue.

5.9.2. Typically, do not analyze more than two items from the same schedule or suspected schedule for each subject or group of subjects unless the analysis of additional items will shift the charge from a misdemeanor to a felony or to a trafficking charge.

5.9.3. Typically, do not analyze misdemeanor items in felony cases.

5.9.4. Do not analyze residues on US currency or stomach contents.

5.9.5. Do not analyze syringes with attached needles unless accompanied by a District Attorney's request.

5.10. Evaluate the number of packages, units or tablets present in an exhibit carefully. If an item to be analyzed contains multiple units other than residues (whether from pipes, baggies, scales, razors, etc.), partially consumed hand-rolled cigarettes or material packaged such that it is impracticable to separate (for analysis purposes, each intact piece of blotter paper is a unit) determine the population. If the item to be analyzed contains a residue or a single package, unit or tablet, proceed to 5.16.

5.10.1. Population Determination

5.10.1.1. Visually inspect each of the packages, units or tablets in the exhibit carefully as well as any contents for uniformity of size, weight, color, packaging, markings, labeling, indications of tampering and other characteristics.

5.10.1.2. If after careful visual inspection it is determined that the packages, units or tablets are uniform, the population shall consist of all of the packages, units or tablets.

5.10.1.3. If there are differences, segregate the packages, units or tablets into individual groups, based upon such observed differences. Analyze each group as separate populations.

5.10.1.4. If in the course of analysis it becomes apparent that the population is not uniform, new populations may be formed based upon individual chemical test results. For hypergeometric sampling, samples which are no longer available for indiscriminate selection may not be considered a part of the new population.

5.10.1.5. If no groups can be formed based upon visual examination, then sampling shall not be performed, proceed to 5.16.

5.11. If the population contains pharmaceutical preparations without indications of tampering, Administrative Sample Selection shall be utilized.

5.12. If the amount of material, dosage units or tablets present does not meet a threshold, Administrative Sample Selection shall be utilized.

5.13. If there is material, dosage units or tablets present in a population to meet a threshold and the individual analysis of the packages, dosage units or tablets is practicable, Threshold Sample Selection shall be utilized.

5.14. If there is material, dosage units or tablets present in a population to meet a threshold and the individual analysis of the packages, dosage units or tablets is not practicable, then the Hypergeometric Sampling Plan shall be utilized.

5.15. Record the sample selection method or sampling plan utilized on the Drug Chemistry worksheet form being used.

5.15.1. Administrative Sample Selection

5.15.1.1. Pharmaceutical Preparations – Initial Submissions, Non-felony/trafficking

5.15.1.1.1. If the physical characteristics do not indicate tampering, sample selection and a chemical analysis are not required.

5.15.1.2. Pharmaceutical Preparations – Resubmission or Felony/Trafficking Chemical Analysis

5.15.1.2.1. If the physical characteristics indicate a controlled substance with no indications of tampering, the complete analysis of one indiscriminately selected unit is required. If the physical characteristics indicate a non-controlled substance, sample selection and chemical analysis are not required.

5.15.1.2.2. For preparations that are weighed, separate weights shall be recorded for the analyzed portion and the unanalyzed portion. Gross weights may be utilized for the unanalyzed portion.

5.15.1.3. Non-pharmaceutical Exhibits

5.15.1.3.1. Analyze a single package, unit or tablet.

5.15.1.3.1.1. In the event that the analysis of the single package, unit or tablet identifies a controlled substance, no further analysis is required.

5.15.1.3.1.2. In the event that the analysis of the single package, unit or tablet does not identify a controlled substance, preliminary testing selected to screen for controlled substances shall be performed on two (or on one for populations consisting of only two) packages, units or tablets. If the

preliminary testing indicates the presence of a controlled substance, complete analysis of that package, unit or tablet is required. If the preliminary testing does not indicate the presence of a controlled substance, no further analysis is required.

5.15.1.3.2. For weighed material, separate weights shall be recorded for the analyzed portion and the unanalyzed portion of the population. Gross weights may be utilized for the unanalyzed portion of the population.

5.15.2. Threshold Sample Selection

5.15.2.1. Refer to the North Carolina Controlled Substances Act and the CCBI Evidence Submission Guide for thresholds. When notified that a submission is intended for prosecution in Federal Courts, refer to the United States Sentencing Commission Guidelines Manual.

5.15.2.2. Perform separate and complete analysis of the number of packages, dosage units or tablets to satisfy the threshold. The net weight minus any uncertainty of measurement should exceed the threshold when possible.

5.15.2.3. For weighed material, separate weights shall be recorded for the analyzed portion and the unanalyzed portion of the population. Gross weights may be utilized for the unanalyzed portion of the population.

5.15.3. Hypergeometric Sampling Plan

5.15.3.1. Perform separate and complete analysis of the number of indiscriminately selected packages, dosage units or tablets as determined from the table below.

5.15.3.1.1. The selection of samples shall be conducted in a manner that prevents the Drug Chemist from consciously selecting a specific unit from the population.

Population Size	Samples
10-11	8
12-13	9
14-15	10
16-17	11
18-20	12
21-23	13
24-26	14
27-30	15
31-34	16
35-39	17
40-45	18

Population Size	Samples
46-52	19
53-61	20
62-73	21
74-88	22
89-108	23
109-138	24
139-184	25
185-270	26
271-474	27
475-1619	28
1620-10000	29

5.15.3.2. To report results with the Hypergeometric sampling plan, the results of analysis to be reported for each individually analyzed unit of a population must be identical and identify the presence of a controlled substance. If non-identical or "no controlled substances" results are obtained, abandon the hypergeometric sampling plan and follow administrative or threshold sample selection.

5.15.3.3. For weighed material, separate weights shall be recorded for the analyzed portion and the unanalyzed portion of the population. Gross weights may be utilized for the unanalyzed portion of the population**.**

5.15.3.4. If there is sufficient material present to satisfy a weight threshold that is not met by the weight of the analyzed portion, obtain individual weights of enough additional indiscriminately chosen samples to meet the weight threshold, if practicable. The net weight minus any uncertainty of measurement should exceed the threshold when possible.

5.15.3.4.1. When the analyzing Drug Chemist determines, based on their training and experience, that it is impracticable to obtain the weight of the required number of individual weights to satisfy a weight threshold, the weight of the population shall be estimated at 95% confidence.

5.15.3.4.2. The Student-t distribution is used to estimate the total weight of the population with a confidence of 95%.

5.15.3.4.2.1. The %RSD (relative standard deviation = 100*(standard deviation of the samples / mean of the samples) must be less than 10 %. Additional samples may be weighed to reach the target percentage. Record the %RSD.

5.15.3.4.2.2. When n/N is greater than 0.1 calculate the estimated total weight as follows using the student-t distribution table below to find the value of t_α. Record the values of n/N, N$\bar{x}$ and $\frac{Nst_\alpha\sqrt{\left(\frac{N-n}{N}\right)}}{\sqrt{n}}$.

$$W = N\bar{x} \pm \frac{Nst_\alpha\sqrt{\left(\frac{N-n}{N}\right)}}{\sqrt{n}}$$

Where:

W = estimated total weight at 95 % confidence
N = number of units in the population
$\bar{x}$ = average weight of the samples (sum of sample weights / n)
x = individual sample weights
n = number of samples
s = standard deviation of the weights of the samples = $\sqrt{\left(\frac{\Sigma(x-\bar{x})^2}{n-1}\right)}$
t_α = the solving value of the Student-t distribution with degrees of freedom, *df* = n-1 within the confidence coefficient α, see table below for values when α = 0.05, confidence = 100%(1 – α) = 95%

5.15.3.4.2.3. When n/N is less than 0.1 calculate the estimated total weight as follows using the student-t distribution table below to find the value of t_α. Record the values of n/N, N$\bar{x}$ and $\frac{Nst_\alpha}{\sqrt{n}}$.

$$W = N\bar{x} \pm \frac{Nst_\alpha}{\sqrt{n}}$$

Where:

W = estimated total weight at 95 % confidence
N = number of units in the population
$\bar{x}$ = average weight of the samples (sum of sample weights / n)
x = individual sample weights
n = number of samples
s = standard deviation of the weights of the samples = $\sqrt{\left(\frac{\Sigma(x-\bar{x})^2}{n-1}\right)}$

t_α = the solving value of the Student-t distribution with degrees of freedom, *df* = n-1 within the confidence coefficient α, see table below for values when α = 0.05, confidence = 100%(1 – α) = 95%

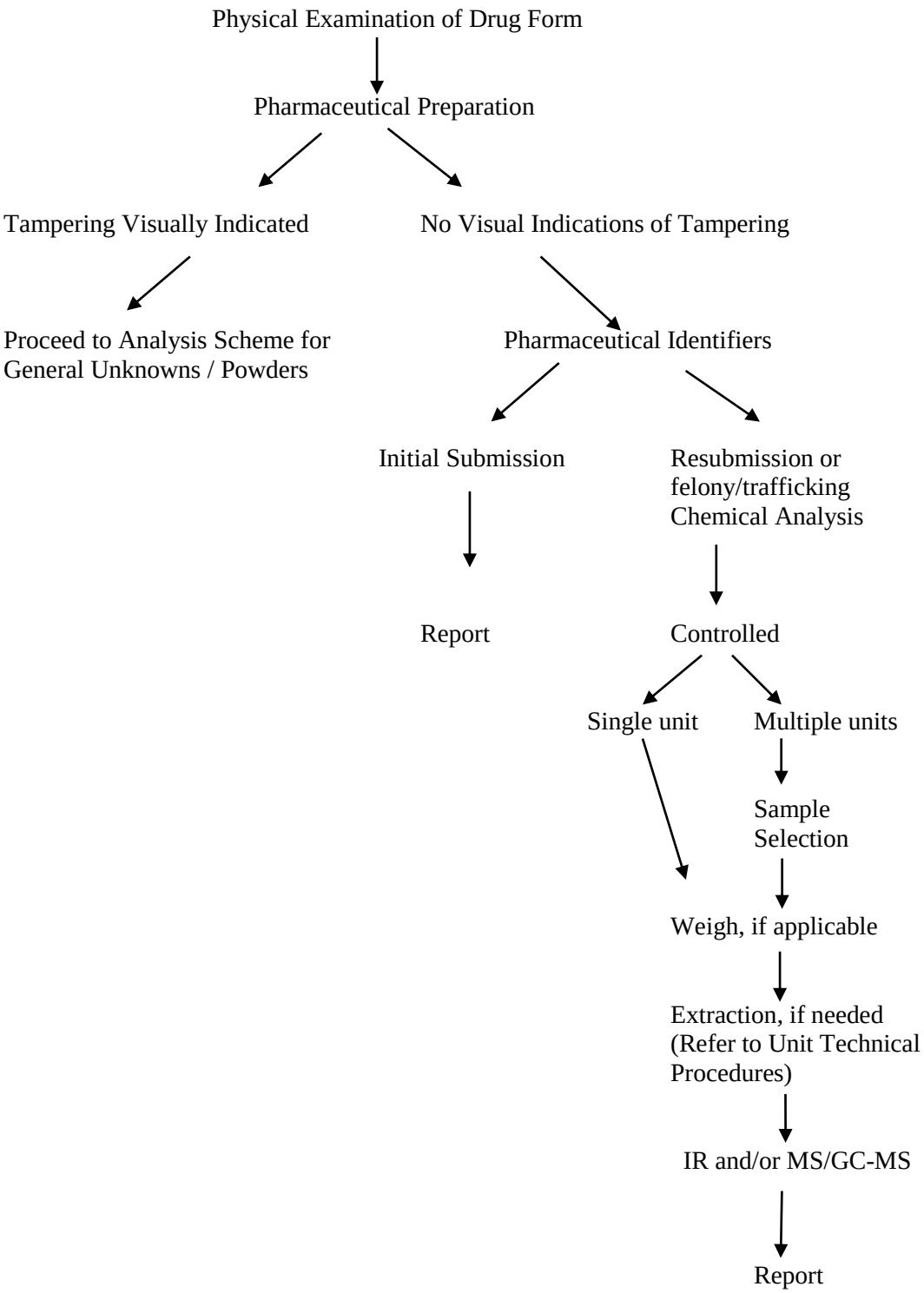
Student-t Distribution	
(t_α values for given degrees of freedom, *df*, with threshold index, α, of 0.05)	
***df* = degrees of freedom = n-1**	$\mathbf{t_\alpha}$ (when α = 0.05)
9	2.262
10	2.228
11	2.201
12	2.179
13	2.160
14	2.145
15	2.131
16	2.120
17	2.110
18	2.101
19	2.093
20	2.086
21	2.080
22	2.074
23	2.069
24	2.064
25	2.060
26	2.056
27	2.052
28	2.048

5.16. Based upon training and experience, determine the most appropriate analysis scheme of the four general analytical schemes for each unit to be analyzed. Perform analyses using only current CCBI Drug Chemistry Unit Technical Procedures.

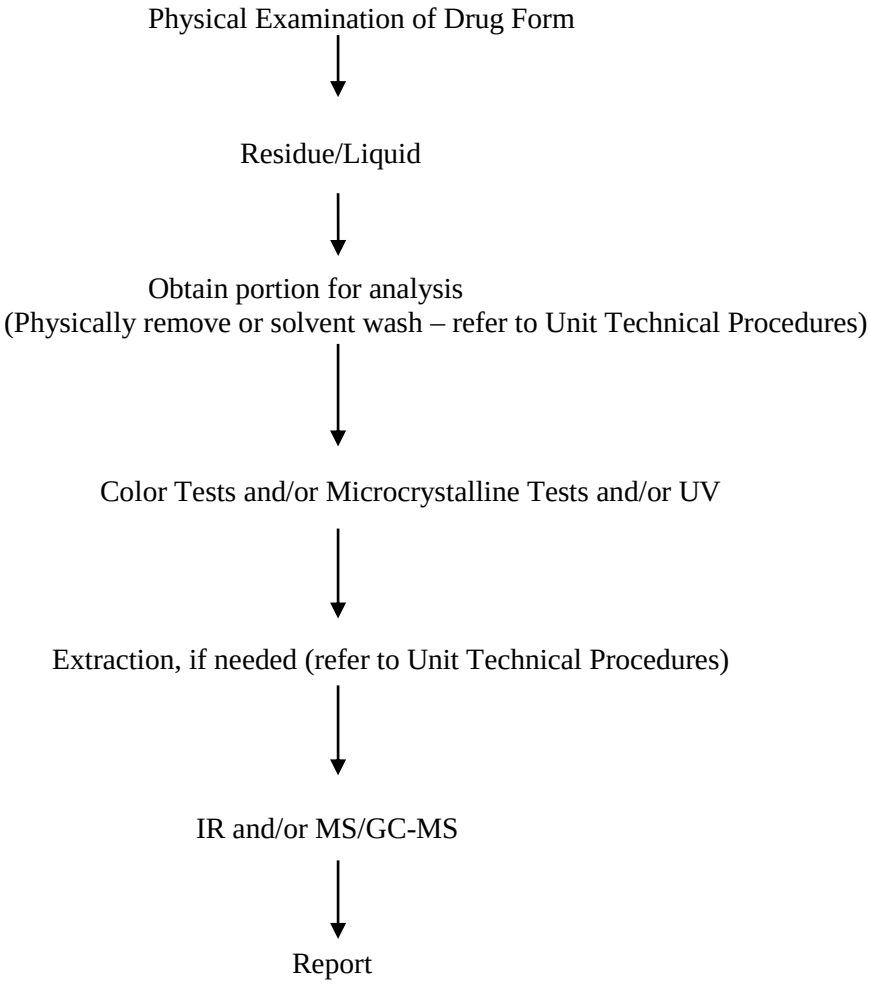
5.16.1. Sample size or other circumstances may require a rearrangement or modification of one or more steps of the analytical schemes.

5.16.2. Use the analytical scheme for general unknowns when a submission is determined by the Drug Chemist to require specialized analysis.

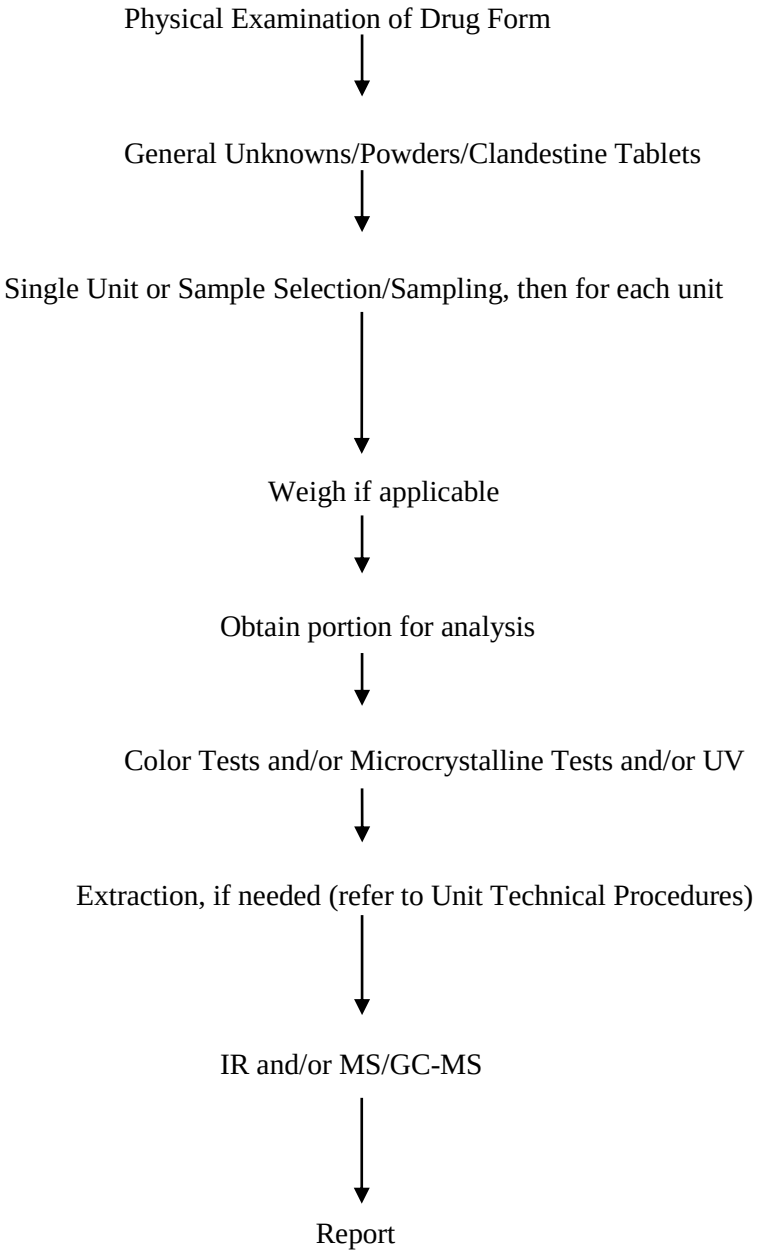
Pharmaceutical Preparation Analysis Scheme



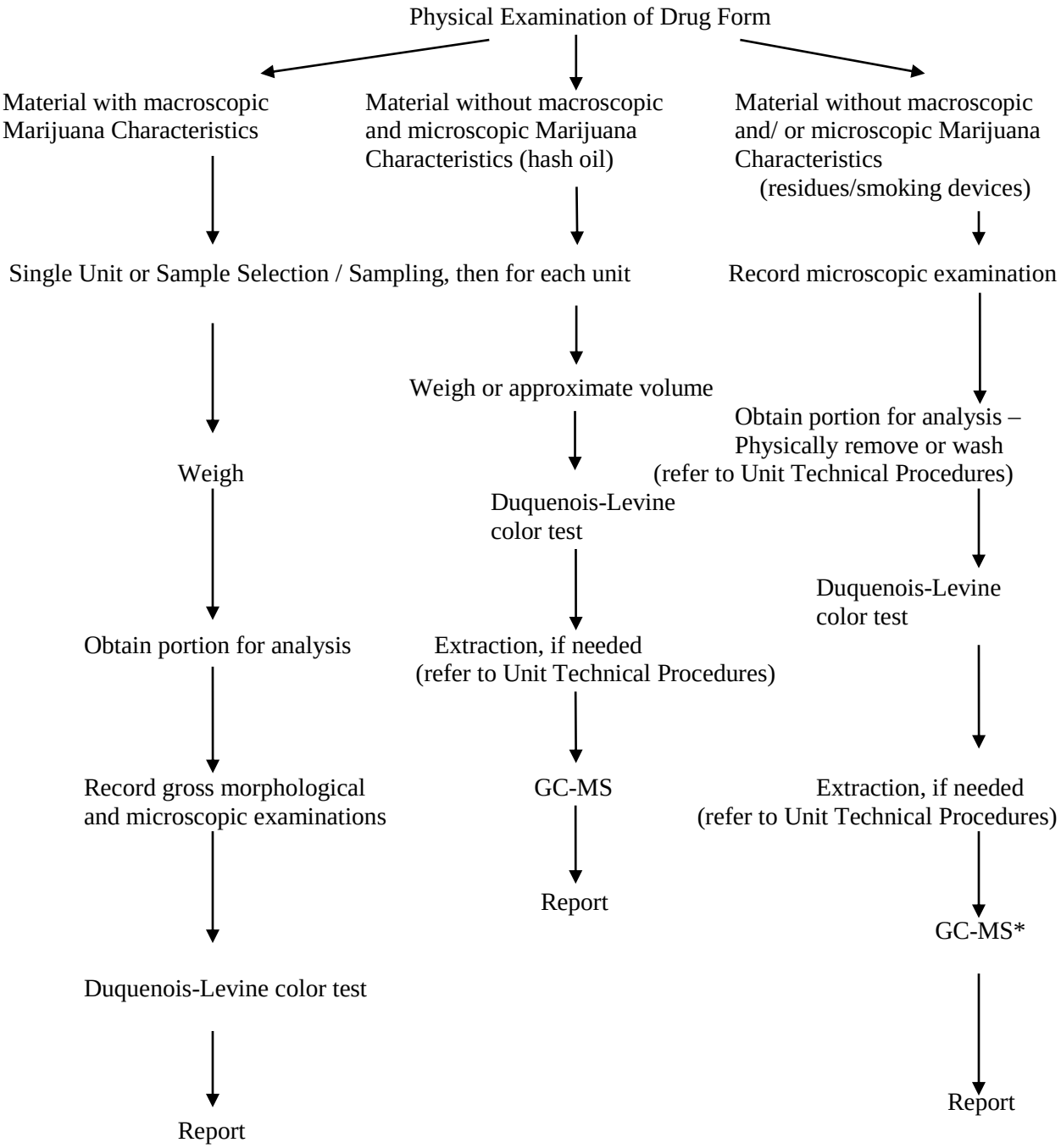
Residue/Liquids Analysis Scheme



General Unknowns/Powders Analysis Scheme



Marijuana Analysis Scheme



* Required if macroscopic characteristics are absent or if another test is inconclusive.

5.17. Minimum Requirements for Identification

5.17.1. Analytical techniques are listed in order of decreasing discriminatory power from Category A to C:

Category A	Category B	Category C
Infrared Spectroscopy	Gas Chromatography	Color Tests
Mass Spectroscopy	Microcrystalline Tests (Not used in conjunction with a Category A Test)	Ultraviolet Spectroscopy
	Pharmaceutical Identifiers	Microcrystalline Tests (Used in conjunction with a Category A Test)
	Cannabis Only: MacroscopicExamination Microscopic Examination (Counts as one each)	

5.17.2. When a Category A technique is incorporated into an analytical scheme, then at least one other technique (from either Category A, B, or C) shall be used for identification of a controlled substance.

5.17.2.1. This combination shall identify the specific drug(s) present, preclude a false positive identification and minimize false negatives.

5.17.2.2. When sufficient material is present, the second technique shall be applied on a separate portion of material. Hyphenated techniques (gas chromatography – mass spectrometry) are considered as separate techniques only when the amount of material present prohibits an additional portion from being obtained.

5.17.2.3. All Category A techniques shall have reviewable data.

5.17.3. When a Category A technique is not used, then at least three different techniques shall be used for identification of a controlled substance.

5.17.3.1. This combination shall identify the specific drug(s) present, preclude a false positive identification and minimize false negatives.

5.17.3.2. Two of the three methods shall be based on uncorrelated techniques from Category B that have reviewable data.

5.17.3.3. A minimum of two separate portions for analysis shall be used in these three tests.

5.17.4. Reviewable data includes:

5.17.4.1. Printed spectra and chromatograms.

5.17.4.2. Reference to published data for pharmaceutical identifiers.

5.17.4.3. Contemporaneous documented peer review, photographs, or digital images of microcrystalline tests if used without a Category A Test.

5.17.4.4. Descriptions of microcrystalline test results, if used in conjunction with a Category A Test.

5.17.4.5. For cannabis and botanical materials only: recording of detailed descriptions of morphological characteristics. Refer to the Drug Chemistry Unit Technical Procedure for the Identification of Marijuana for descriptions used in conjunction with the Drug Chemistry Worksheet or Drug Chemistry Blank Worksheet.)

5.17.5. For the use of any method to be considered of value in the identification of the controlled substance, the test shall be considered positive. While negative tests provide useful information for ruling out the presence of a particular drug or drug class, these results have no value toward establishing the positive identification of a drug.

5.17.6. Macroscopic and microscopic examination of cannabis shall be considered as two separate Category B techniques when observations include documented botanical features as described in the Drug Chemistry Unit Technical Procedure for Identification of Marijuana.

5.17.7. For Cannabis related exhibits that lack observable macroscopic and microscopic botanical detail, i.e., extracts and residues, any controlled substances shall be identified utilizing the principles in Units **5.17.2** or **5.17.3** set forth in this procedure.

5.18. Analytical Techniques

5.18.1. Color tests may be used to screen for the presence of controlled substances or aid in the identification of a controlled substance as a Category C test. Refer to the Drug Chemistry Unit Technical Procedure for Color Tests.

5.18.1.1. Choose color tests based upon usefulness, i.e., modified sodium nitroprusside for methamphetamine, Marquis for heroin.

5.18.2. Ultraviolet spectroscopy may be used to screen for the presence of controlled substances or aid in the identification of a controlled substance as a Category C test. UV may be performed on extracted submitted material or on straight submitted material if it does not

contain substances that interfere with the analyte of interest. Refer to the Drug Chemistry Unit Technical Procedure for Ultraviolet Spectroscopy.

5.18.2.1. Ultraviolet spectroscopy may be used for quantitative comparisons in cases involving dilution/diversion.

5.18.3. Microcrystalline tests may be used to screen for the presence of controlled substances or aid in the identification of a controlled substance as a Category B or Category C test. Refer to the Drug Chemistry Unit Technical Procedure for Microcrystalline Tests.

5.18.3.1. When a microcrystalline test is used as a Category C test, i.e., in conjunction with a Category A test, a description of the crystals shall be recorded on the Drug Chemistry worksheet form being used.

5.18.3.2. When a microcrystalline test is used as a Category B test, i.e., not in conjunction with a Category A test, the crystals shall be contemporaneously peer reviewed by another Drug Chemist. Document the peer review on the Drug Chemistry worksheet form being used, refer to 5.17.4.

5.18.4. Pharmaceutical Identifiers - The markings and physical characteristics of pharmaceutical preparations may be used to determine the consistency of units, to screen for the presence of controlled substances or aid in the identification of a controlled substance as a Category B test. For the pharmaceutical identifiers to be considered of value in the identification of the controlled substance there must not be any indications of tampering. Carefully inspect and record the markings and physical characteristics, including shape and color. Record any indications of tampering. Use only credible reference materials, i.e., *Micromedex*, *The Physician's Desk Reference, The Logo Index for Tablets and Capsules* and manufacturer's published data.

5.18.5. Extractions – Extractions may be used to isolate controlled substances from mixtures for analysis and / or prepare material for analysis techniques. Refer to the Drug Chemistry Unit Technical Procedure for Extractions.

5.18.6. Infrared Spectroscopy may be used to screen for the presence of controlled substances, determine the salt form of a controlled substance and aid in the identification of a controlled substance as a Category A test, refer to the Drug Chemistry Unit Technical Procedure for Infrared Spectroscopy.

5.18.6.1. IR may be used as a Category A test only when the spectrum of submitted material, straight or extracted, has a positive comparison to primary or secondary reference material.

5.18.6.2. IR may be used to determine the salt form of a controlled substance only when the areas of the spectrum necessary to identify the salt form have a positive comparison to primary or secondary reference material.

5.18.7. Gas Chromatography - Mass Spectrometry may be used to screen for the presence of controlled substances in properly prepared materials. Gas chromatography may be used to aid in the identification of a controlled substance as a Category B test. Mass Spectrometry may be used to aid in the identification of a controlled substance as a Category A test. Refer to the Drug Chemistry Unit Technical Procedure for Gas Chromatography - Mass Spectrometry and the Drug Chemistry Unit Technical Procedure for Extractions.

5.18.7.1. Gas chromatography may be used to aid in the identification of a controlled substance as a Category B test only when the retention time of a of submitted material, straight or extracted, has a positive comparison to primary or secondary reference material, refer to the Drug Chemistry Unit Technical Procedure for Gas Chromatography - Mass Spectrometry.

5.18.7.2. Mass Spectrometry may be used to aid in the identification of a controlled substance as a Category A test only when the mass spectrum of a submitted material, straight or extracted, has a positive comparison to primary or secondary reference material, refer to the Drug Chemistry Unit Technical Procedure for Gas Chromatography – Mass Spectrometry.

5.19. Record the weight received and returned of analyzed material other than residues and liquids less than approximately one milliliter, refer to the Drug Chemistry Unit Technical Procedure for Balances.

5.20. For each unit to be analyzed, obtain the material for analysis. Although visual homogeneity is considered when obtaining the material for analysis, no assumption about homogeneity of the material is made for reporting purposes, i.e., results are reported as "found to contain."

5.20.1. If the material is visually homogenous, obtain the portion needed for analysis.

5.20.1.1. For larger, compressed, visually homogenous materials (e.g., kilos of suspected cocaine, bricks of suspected marijuana) obtain multiple portions of the material from different areas and combine for analysis. Select a number of portions appropriate to allow portions to be collected throughout the material. Record the number and location of the portions.

5.20.2. If the material is not visually homogenous, perform one of the following:

5.20.2.1. Render the material homogenous by crushing, grinding, etc. Record the technique used to render the material homogenous. Obtain a portion of the homogenous material for analysis.

5.20.2.2. Obtain a portion of each type of material present and analyze individually. Record the number and location of the portions collected.

5.20.3. If the material is a residue a portion for analysis may be obtained by physically removing a portion by scraping or by performing a wash with a suitable solvent.

5.20.4. If the material is a liquid, mix thoroughly and observe for any layering as the liquid settles. If only a single layer is observed, remove an aliquot for analysis. If multiple layers are observed, obtain an aliquot of each layer and analyze individually. Use pipettes and / or additional containers to separate the layers and record the technique used for separation.

5.21. All Drug Chemistry submissions analyzed shall be reported on a Drug Chemistry Unit Report, refer to the CCBI report writing manual. While Chemists may utilize self-prepared templates containing commonly used report wording for use in preparation of reports, the templates shall not contain case specific information such as weights, case numbers, dates, etc.

5.21.1. The report shall include the submitting agency item number(s), a detailed description of the item(s) including the number of units or dosage units and the condition of the item(s) for each item submitted for Drug Chemistry analysis in the "Item(s) Submitted" field.

5.21.2. The report shall include "Controlled Substances" in the "Type Analysis Requested" field.

5.21.3. The report shall include a "Disposition" field. The disposition of the submitted items shall be stated in this field.

5.21.4. The report shall include a "Results and Conclusions" field. The results and conclusions of each analysis shall be included in this field along with the associated submitting agency item number(s).

5.21.4.1. Items which are not analyzed shall be included in this field along with the associated submitting agency item number(s) followed by "No analysis."

5.21.4.2. When a net weight of analyzed material is reported, include the expanded uncertainty and the coverage probability. Refer to the Drug Chemistry Unit Technical Procedure for Uncertainty of Measurement.

5.21.4.3. For substances which are only federally controlled, modify the report statement to include: *"a federally controlled substance, (insert schedule) according to 21 CFR § 1308 and 21 USC §801 et seq."*

5.21.5. Results and Conclusions Reporting for Single Unit Items and Initial Submissions of Non-felony/trafficking Pharmaceutical Preparations

5.21.5.1. The result for a unit found to contain identified controlled substance(s) shall contain the item number, *"Found to contain:"* followed by the name(s) of the substance(s) and the assigned schedule of the substance according to the current North Carolina General Statutes.

5.21.5.1.1. If a net weight was recorded, the results shall be followed by *"Net weight of the (insert description):"* followed by the net weight of the material.

5.21.5.1.2. If the material was recorded to be a residue, an amount of material which could not be readily removed from the container in which it was submitted, include *"Residue Amount."*

5.21.5.2. For multiple units with identical results, identify the analyzed portion in the "Results and Conclusions" section of the Laboratory Report with the submitting agency item number, any additional information required for unique identification and the following statement

"(insert number of packages, units or tablets)(insert description) were individually analyzed and were each found to contain" followed by the identity of the controlled substance(s) identified and the assigned schedule of the substance according to the current North Carolina General Statutes.

Include the weight with the following statement:

"Net weight of the (insert description):" followed by the net weight of the material.

5.21.5.3. The result for a unit in which a controlled substance was not identified shall be reported as *"No controlled substances."*

5.21.5.3.1. If a net weight was recorded, the results shall be followed by *"Net weight of the (insert description):"* followed by the net weight of the material.

5.21.5.3.2. If the material was recorded to be a residue, an amount of material which could not be readily removed from the container in which it was submitted, include *"Residue Amount."*

5.21.5.4. The result for an initial submission consisting of a pharmaceutical preparation (non-felony/trafficking amount) found to be consistent with a controlled preparation that has not been tampered with may be reported as follows:

"The physical characteristics, including shape, color and manufacturer's markings of the (insert description), were visually examined and found to be consistent with a pharmaceutical preparation that contains:" followed by the identity of the controlled substance(s) identified and the assigned schedule according to the North Carolina General Statues, if applicable.
There were no visual indications of tampering.
No chemical analysis was performed."

For weighed material include the weight with the following statement:

"Net weight of the (insert description):" followed by the net weight of the material.

5.21.5.5. The result for a submission consisting of a pharmaceutical preparation found to be consistent with a non-controlled preparation that has not been tampered with shall be reported as follows:

"*The physical characteristics, including shape, color and manufacturer's markings of the unit, were visually examined and found to be consistent with a pharmaceutical preparation that does not contain a controlled substance.*
There were no visual indications of tampering.
No chemical analysis was performed."

5.21.5.6. The result for a unit that did not contain a sufficient amount of material for a complete analysis shall be reported as:

"Insufficient material for analysis."

5.21.6. Results and Conclusions Reporting for Multi-Unit Items

5.21.6.1. Reporting Identified Substances – Administrative Sample Selection – Pharmaceutical Preparation

5.21.6.1.1. Each population shall be sufficiently described in the "Items Submitted" section of the Laboratory Report to substantiate the grouping of the preparations into the population.

5.21.6.1.2. The analyzed portion shall be identified in the "Results and Conclusions" section of the Laboratory Report with the submitting agency item number, any additional information required for unique identification and the following statement:

"One (insert description) was analyzed and found to contain" followed by the identity of the controlled substance(s) identified, the assigned schedule according to the North Carolina General Statutes, if applicable.

Include the weight with the following statement:

"Net weight of the (insert description):" followed by the net weight of the material.

5.21.6.1.3. The unanalyzed portion of the population shall be identified in the "Results and Conclusions" section of the Laboratory Report with the following statement

"(insert number of packages, units or tablets)(insert description) (was/were) visually examined; however, no chemical analysis was performed."

If applicable, include the weight with the following statement

"(insert Net or Gross) Weight of (insert description) – followed by the net or gross weight of the material.

5.21.6.1.4. Include the following statement in the "Results and Conclusions" section of the Laboratory Report on the line directly below the line generated in **5.21.6.1.3.**

"The physical characteristics, including shape, color and manufacturer's markings of all (insert description) were visually examined and found to be consistent with a pharmaceutical preparation containing (insert substance(s) indicated and schedule(s)). There were no visual indications of tampering."

5.21.6.2. Reporting Identified Substances – Threshold Sample Selection and Administrative Sample Selection with Non-Pharmaceuticals

5.21.6.2.1. Each population shall be thoroughly described in the "Items Submitted" section of the Laboratory Report to substantiate the grouping of the packages, units or tablets into the population.

5.21.6.2.2. For each portion of the population with identical results, identify the analyzed portion in the "Results and Conclusions" section of the Laboratory Report with the submitting agency item number, any additional information required for unique identification and the following statement

"(insert number of packages, units or tablets)(insert description) were individually analyzed and were each found to contain" followed by the identity of the controlled substance(s) identified and the assigned schedule of the substance according to the current North Carolina General Statutes.

Include the weight with the following statement:

"Net weight of the (insert description):" followed by the net weight of the material.

5.21.6.2.3. The unanalyzed portion of the population shall be identified in the "Results and Conclusions" section of the Laboratory Report with the following statement

"(insert number of packages, units or tablets)(insert description): "No chemical analysis was performed."

Include the weight with the following statement:

"(insert Net or Gross) weight of (insert description) – followed by the net or gross weight of the material.

5.21.7. Reporting Identified Substances – Hypergeometric Sampling Plan

5.21.7.1. Each population shall be sufficiently described in the "Items Submitted" section of the Laboratory Report to substantiate the grouping of the packages, units or tablets into the population.

5.21.7.2. The analyzed portion shall be identified in the "Results and Conclusions" section of the Laboratory Report with the submitting agency item number, any additional information required for unique identification and the following statement

"(insert number of packages, units or tablets)(insert description) were individually analyzed and were each found to contain" followed by the identity of the controlled substance(s) and the assigned schedule of the controlled substance according to the North Carolina General Statutes, if applicable.

Include the weight of the analyzed portion with the following statement:

"Net weight of (insert description):" followed by the weight of the material.

5.21.7.3. The unanalyzed portion shall be identified in the "Results and Conclusions" section of the Laboratory Report with the following statement (if the unanalyzed portion contains un-weighed material, it shall be listed separately from any weighed material)

"(insert number of packages, units or tablets)(insert description): "No chemical analysis was performed."

If applicable, include the weight with the following statement

"(insert Net or Gross) weight of (insert description):" followed by the weight of the material.

5.21.7.4. Include the following statement

"This material was analyzed with a statistical sampling plan that demonstrates with 95 % confidence that at least 90 % of the individual units contain the identified substance(s)."

5.21.7.5. If the total weight of the population is estimated, the estimated weight shall be identified in the "Results of Examination" section of the Laboratory Report with the following statement:

"(insert number of packages, units or tablets)(insert description)" were individually weighed. The total weight of the (insert total number of packages, units or tablets in the population) (insert description) was estimated with 95% confidence to be (insert estimated total weight of the population, $N\bar{x}$) ± (insert estimated weight adjustment for 95% confidence, either $\frac{Nst_{\alpha}\sqrt{\left(\frac{N-n}{N}\right)}}{\sqrt{n}}$ or $\frac{Nst_{\alpha}}{\sqrt{n}}$.)

5.21.8. Reporting Non-Controlled Substances – Administrative Sample Selection – Pharmaceutical Preparations

5.21.8.1. Each population shall be thoroughly described in the "Items Submitted" section of the Laboratory Report to substantiate the grouping of the preparations into the population.

5.21.8.2. The population shall be identified in the "Results and Conclusions" section of the Laboratory Report with the item number, any additional information required for unique identification and the following statement

"The physical characteristics, including shape, color and manufacturer's markings of all (insert description) were visually examined and found to be consistent with a pharmaceutical preparation that does not contain a controlled substance.
There were no visual indications of tampering.
No chemical analysis was performed."

5.21.9. Reporting Non-controlled Substances – Hypergeometric Sampling, Threshold Sample Selection and Administrative Sample Selection with Non-Pharmaceuticals

5.21.9.1. Each population shall be thoroughly described in the "Items Submitted" section of the Laboratory Report to substantiate the grouping of the packages, units or tablets into the population.

5.21.9.2. The portion subjected to complete analysis shall be identified in the "Results and Conclusions" section of the Laboratory Report with the submitting agency item

number, any additional information required for unique identification and the following statement:

"(insert number of packages, units or tablets)(insert description) (was / were) individually analyzed and (was / were) not found to contain a controlled substance."

Include the weight with the following statement:

"(insert Net or Gross) weight of (insert description) – followed by the net or gross weight of the material.

5.21.9.3. The portion subjected to preliminary testing shall be identified in the "Results and Conclusions" section of the Laboratory Report with the following statement:

"(insert number of packages, units or tablets)(insert description) were individually subjected to preliminary testing that did not indicate the presence of a controlled substance."

Include the weight with the following statement

"(Net or Gross) weight of (insert description) – followed by the net or gross weight of the material.

5.21.9.4. The unanalyzed portion shall be identified in the "Results of Examination" section of the Laboratory Report with the following statement

"(insert number of packages, units or tablets)(insert description): No chemical analysis."

If applicable, include the weight with the following statement

"(Net or Gross) weight of (insert description) – followed by the net or gross weight of the material.

5.21.9.5. No statistical inferences shall be made.

5.22. Review

5.22.1. All cases shall be subjected to administrative and technical review prior to the release of the report.

5.22.2. The reviews shall be performed in accordance with the CCBI Crime Laboratory Administrative Procedure for Case File Review.

5.22.3. Technical Review

5.22.3.1. The technical review shall be performed by a Drug Chemist other than the analyzing Drug Chemist.

5.22.3.2. The technical review shall include a review of the report and all examination records to ensure:

5.22.3.2.1. Appropriate analyses have been performed and are in conformance with CCBI Drug Chemistry Unit policies and procedures as well as CCBI Crime Laboratory policies and procedures.

5.22.3.2.2. Calculations and data transfers are accurate.

5.22.3.2.3. The conclusions of the analyzing Drug Chemist are reasonable, supported by the examination records and within the constraints of validated scientific knowledge.

5.22.3.2.4. The report is clear, accurate and complete.

5.22.3.3. The Technical Reviewer shall document the review on the Drug Chemistry Case Notes Coversheet.

5.22.3.3.1. Record any comments in the Review Comments section and initial and date the form.

5.22.3.3.2. If no discrepancies are observed and all requirements in 5.22.3.2. are met, mark the Status as approved. The case is ready for administrative review, refer to 5.22.4.

5.22.3.3.3. If discrepancies are observed and / or all requirements in 5.22.3.2. are not met:

5.22.3.3.3.1. Record all discrepancies in the Review Comments section.

5.22.3.3.3.2. Initial and date the form.

5.22.3.3.3.3. Mark the Status as returned.

5.22.3.3.3.4. Return the case to the analyzing Drug Chemist. Report any significant discrepancies to the Crime Laboratory Deputy Director.

5.22.3.3.3.5. The analyzing Drug Chemist shall make any necessary changes according to the Crime Laboratory Quality Assurance Manual and submit the case for another technical review to the same Technical Reviewer.

5.22.3.3.3.6. If a conflict arises between the analyzing Drug Chemist and the Technical Reviewer that cannot be resolved through discussion and review of procedures and literature, the Drug Chemistry Technical Leader and the Crime Laboratory Deputy Director shall be notified.

5.22.3.3.3.6.1. The Drug Chemistry Technical Leader and the Crime Laboratory Deputy Director shall determine a resolution. The resolution shall be communicated to the analyzing Drug Chemist and the Technical Reviewer and documented in the examination records.

5.22.4. Administrative Review

5.22.4.1. The administrative review shall include:

5.22.4.1.1. A review of the report for spelling and grammatical accuracy.

5.22.4.1.2. A review of all administrative and examination records to ensure that the records are uniquely identified according to CCBI policy and procedure.

5.22.4.1.3. A review of the report to ensure that it is clear, accurate and complete.

5.22.4.1.4. A review of the Drug Chemistry Case Notes Coversheet to ensure that the technical review has been completed and approved.

5.22.4.2. The administrative review shall be performed by either the Crime Laboratory Deputy Director or a Drug Chemist other than the analyzing Drug Chemist.

6. Limitations – Refer to the Drug Chemistry Unit technical procedures.

7. Safety – Refer to the CCBI Crime Laboratory Safety Manual.

8. References

8.1.1. ASTM Standard E2329-14 ~~09~~. "Standard Practive for Identification of Seized Drugs." ASTM International: West Conshohocken, PA, 2014 ~~2009~~, www.astm.org.

8.1.2. "Part III B – Methods of Analysis/Drug Identification." *Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG) Recommendations.* 7th ~~5th~~ Edition. August 14, 2014 ~~January 29, 2010~~.

8.1.3. *Guidelines on Representative Drug Sampling*. United Nations, New York: United Nations Office on Drugs and Crime, 2009.

8.1.4. Frank, Richard S., et. al. "Representative Sampling of Drug Seizures in Multiple Containers." *Journal of Forensic Sciences*, Volume 36, Issue 2 (March 1991), 350-357.

8.1.5. "PART III A - Methods of Analysis/Sampling Seized Drugs for Qualitative Analysis." *Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG) Recommendations.* 7th ~~5th~~ ed.: August 14, 2014 ~~January 29, 2010~~.

9. Records

9.1. Drug Chemistry Cover Sheet form
9.2. Drug Chemistry Worksheet form
9.3. Drug Chemistry Additional Page Worksheet form
9.4. Drug Chemistry Additional Weight Worksheet form
9.5. Drug Chemistry Hypergeometric Weight Estimation Worksheet form
9.6. Drug Chemistry Summed Weights form
9.7. Weekly Cleaning Log form

Revision History		
Effective Date	Version Number	Reason
1/1/13	1	Compliance with ASCLD/LAB requirements
8/7/13	2	Incorporation of Uncertainty of Measurement and reduce Administrative Sampling
7/14/14	3	Include use of Agency item numbers, ref LAPM 14, and addition of abbreviation "ow"
2/16/15	4	Additions to section 5.3 and line 5.15.2.1. Addition of weekly cleaning log to records section 9.
3/31/16	5	Addition to 5.21. Remove stricken text in 5.15.3.4.2.2. Insert ending parenthesis in 5.21.7.5. Updated references 8.1.1, 8.1.2 and 8.1.5.

2: Technical Procedure for the Identification of Marijuana

1. Purpose/Scope - This procedure provides direction for the identification of Marijuana as defined in NC General Statute §90-87 (16) and Hashish as defined in §90-95(d)(4) in the Drug Chemistry Unit of the Raleigh/Wake City-County Bureau of Identification.

2. Definitions

2.1. Reference material – Material sufficiently homogeneous and stable with reference to specified properties, which has been established to be fit for its intended use in measurement or in examination of nominal properties.

3. Abbreviations

3.1. Refer to the Drug Chemistry Unit Technical Procedure for Drug Chemistry Analysis
3.2. Macro – macroscopic
3.3. Micro – microscopic
3.4. Cys – cystolithic
3.5. Gld – glandular
3.6. Tri – trichome

4. Equipment, Materials and Reagents

4.1. Equipment

4.1.1. Nikon SMZ645 stereomicroscope equipped with 10X eyepiece and 0.8X to 5X zoom capability to produce magnification of 8-50X
4.1.2. Nikon Eclipse E400 Pol polarizing microscope equipped with 10X eyepiece and 10X objective to produce magnification of 100X

4.2. Materials and Reagents

4.2.1. Marijuana reference material
4.2.2. Chloroform, ACS grade

5. Procedure

5.1. Refer to the Drug Chemistry Analysis Technical Procedure for sampling.

5.2. For exhibits with a gross weight of less than 5 grams consisting of hand-rolled cigarettes or partial hand-rolled cigarettes, the paper may be included in the weight recorded and reported. The evidence may be cut open to expose the plant material for viewing and analysis. Use the following statement to report the weight when the paper is included:

"Weight of paper and plant material:"

5.3. Observe plant material macroscopically and microscopically to verify the presence of visually recognizable morphological characteristics using marijuana reference material for comparison.

5.4. Record the lot number or Drug Chemistry designation of the reference material used for comparison on the Drug Chemistry Worksheet form.

5.5. Macroscopic characteristics – Record the observed macroscopic characteristics present in the exhibit on the Drug Chemistry Worksheet form. References to line numbers of this procedure may be used. Include any additional details as needed.

5.5.1. An exhibit must contain sufficient macroscopic characteristics described and referenced in this procedure to be macroscopically consistent with marijuana or be visually consistent with marijuana reference material for the macroscopic examination to be considered as a positive Category B test, refer to the Drug Chemistry Drug Analysis Technical Procedure.

5.5.2. Macroscopic Characteristics

5.5.2.1. Upright stalk attains a height of 3-16 feet, usually 4-6 feet.
5.5.2.2. Stalk varies in diameter up to two inches, usually one-half inch or less.
5.5.2.3. Stalks and stems are longitudinally grooved.
5.5.2.4. Nodes occur on the stalk at intervals of 4 to 20 inches. The plant branches at the nodes – a branch appearing immediately above each leaf. The branches occur at opposite points on the stalk with alternate pairs situated at approximately right angles except at the top of the plant, where the arrangement becomes alternate rather than opposite.
5.5.2.5. Plant has compound palmate leaves with 5-11 leaflets (usually seven), and odd in number.
5.5.2.6. Leaflets are pointed at both ends and vary up to about 6 inches length and to about 1.5 inches in width. They are characteristically hair covered, veined and serrated (with notched edges.)
5.5.2.6.1. The veins run out obliquely from the midrib to the tips of the teeth.
5.5.2.6.2. The teeth point towards the tips.
5.5.2.6.3. The upper surface is darker than the lower surface.
5.5.2.7. Distinction between male and female plants is difficult except at maturity.
5.5.2.7.1. Male: flowers are very prominent; mature ones shed pollen profusely.
5.5.2.7.2. Female: flowers are inconspicuous and are found hidden among the small leaves at the ends of the stalk and branches.
5.5.2.8. Seeds are about 2 – 5 mm long, greenish-yellow to brown, mottled, covered with lacy markings, ovoid in shape and divided into two segments by a ridge extending around the greatest circumference.
5.5.2.9. Seeds are enclosed in hulls or pods which are green, hairy and sticky to the touch.
5.5.2.10. Seeds contain a white, oily, meaty substance similar to coconut meat.

5.5.2.11. The root system consists of one main tap root up to eight inches long, from which spring a number of comparatively tiny branches.

5.5.2.12. Plant has a characteristic odor and is sticky to the touch.

5.6. Microscopic Characteristics

5.6.1. An exhibit must contain leaves that meet the requirements of 5.6.2.3.3 and 5.6.2.3.4. for the microscopic examination to be considered as a positive Category B test, refer to the Drug Chemistry Drug Analysis Technical Procedure.

5.6.2. Microscopic Characteristics - Observe the microscopic characteristics using the stereomicroscope and record the observations on the Drug Chemistry Worksheet form by checking the box beside the characteristics, if applicable, or recording a written description of the observation. References to line numbers of this procedure may be used. Include any additional details as needed.

5.6.2.1. The plant has glandular (related to a cell or group of cells that produces a secretion) trichomes (hair-like projections) where the cannabis resin is produced and stored. They are mainly associated with the flower structures but they can also be found on the lower surface of the leaves and occasionally on the stems of young plants. They occur as:

5.6.2.1.1. Sessile glands, i.e. trichomes without stalk

5.6.2.1.2. Small bulbous glandular trichomes with one-celled stalks

5.6.2.1.3. Long multicellular stalks on female flowers

5.6.2.2. The plant has non-glandular trichomes which are unicellular, rigid and curved with a slender pointed apex.

5.6.2.3. Required characteristics for identification of marijuana leaves:

5.6.2.3.1. Green, brown or brown-spotted in color

5.6.2.3.2. Characteristically veined and serrated, refer to 4.5.2.6.1 – 4.5.2.6.2.

5.6.2.3.3. Non-glandular cystolithic hairs on the upper side with a characteristic bear claw shape with cystoliths, calcium carbonate crystals, visible at their bases. Some hairs may be broken and the cystolith freed. Dilute hydrochloric acid may be added to produce effervescence with the calcium carbonate cystolith

5.6.2.3.4. Non glandular, non-cystolithic hairs on the lower surface which are longer, more slender and more sharply pointed than the hairs on the upper surface.

5.6.2.4. Required characteristics for identification of marijuana stems

5.6.2.4.1. Green, brown or brown-spotted in color

5.6.2.4.2. Longitudinally grooved.

5.6.2.5. Required characteristics for identification of marijuana seeds**:**

5.6.2.5.1. Greenish-yellow to brown, mottled
5.6.2.5.2. Covered with lacy markings
5.6.2.5.3. Ovoid in shape
5.6.2.5.4. Ridge around the greatest circumference
5.6.2.5.5. Contain a white, oily, meaty substance similar to coconut meat

5.6.2.6. Required characteristics for identification of marijuana hulls

5.6.2.6.1. Green, brown or brown-spotted in color
5.6.2.6.2. Characteristically shaped, ovoid
5.6.2.6.3. Non-glandular cystolithic hairs on outer surface
5.6.2.6.4. Glandular hairs which are shaped like clubs with flattened spherical heads

5.7. Hashish, the extracted resin of marijuana - Observe the microscopic characteristics using the polarizing microscope, the sample may be wetted with a drop of chloroform, and record the observations on the Drug Chemistry Worksheet form.

5.7.1. Hashish contains the resinous secretions of the Marijuana plant as well as finer plant material. It appears as loose or pressed sticky powder, depending on the method of production.

5.7.2. Powder material to be reported to contain Δ^9-Tetrahydrocannabinol that also has microscopic plant characteristics may additionally be identified on the report as "Hashish."

5.7.2.1. Examples of hashish microscopic characteristics: leaf fragments, hairs.

6. Limitations

6.1. Morphological characteristics and variation in color of cannabis plants are influenced by the seed strain as well as by environmental factors such as light, water, nutrients and space.

6.2. Not every marijuana exhibit contains every plant characteristic. The Drug Chemist shall identify and document those that are present.

6.3. Immature seedlings may not exhibit sufficient morphological characteristics for identification.

7. Safety

7.1. Use proper personal protective equipment when handling moldy marijuana.
7.2. Refer to the CCBI Crime Laboratory Safety manual.

8. References

8.1. *Marihuana Its Identification*. Washington, D.C.: U.S. Treasury Department Bureau of Narcotics, United States Printing Office, 1948.

8.2. *Recommended Methods for the Identification and Analysis of Cannabis and Cannabis Products.* New York: Laboratory and Scientific Section United Nations Office on Drugs and Crime, United Nations, 2009.

8.3. *North Carolina General Statutes* §90-87 (16) and §90-95(d)(4).

Revision History		
Effective Date	Version Number	Reason
1/1/2013	1	ISO Compliance
2/16/2015	2	Corrected numbering throughout

3: Technical Procedure for General Laboratory Equipment

1. **Purpose / Scope** - This procedure provides direction for the use of general laboratory equipment in the Drug Chemistry Unit of the Raleigh/Wake City-County Bureau of Identification.

2. **Definitions –** N/A

3. **Abbreviations**

   3.1. Refer to the Drug Chemistry Unit Technical Procedure for Drug Chemistry Analysis

4. **Equipment**

   4.1. Refrigerators
   4.2. Microscopes
   4.3. Laboratory environmental monitor – barometer, hygrometer, thermometer, NIST traceable
   4.4. Millipore Elix Advantage Water Purification System with E-POD Unit

5. **Materials**

   5.1. Millipore Elix Advantage Progard Pack
   5.2. Millipore Elix Advantage Reservoir Vent Filter
   5.3. Millipore Elix Advantage UV 254 lamp
   ~~**5.4.** Millipore Elix Advantage POD Pak~~
   5.5. Millipore Elix Advantage chlorine tablet

6. **Procedure for Microscopes**

   6.1. New microscopes shall be installed by an approved vendor and subjected to performance verification according to the CCBI Crime Laboratory Administrative Procedure for Validation and Performance Verification.

   6.2. Microscopes should be turned off and covered when not in use.

   6.3. Replace microscope lamps as needed. Record the service in the microscope log.

   6.4. Microscopes will be serviced annually by an approved vendor. The service will include cleaning, lubricating and alignment. Record the service in the microscope log.

   6.5. If the amount of light passing through the optics decreases significantly so that a sample cannot be seen, place the microscope out of service and notify the Drug Chemistry Technical Leader for service scheduling.

6.6. See references for manufacturer's instructions.

6.7. Microscope logs will be maintained in the Drug Chemistry Unit.

7. Laboratory environmental monitor

7.1. The building environmental controls generally provide adequate laboratory environmental conditions for analysis. When environmental conditions are outside of acceptable levels they are apparent to a Drug Chemist based on their sensory perception of the environment. When a Drug Chemist perceives an abnormally high or low temperature or abnormally high humidity level the Drug Chemist shall:

- Notify General Services Administration that there is an environmental control problem and provide the applicable room number
- Notify the Drug Chemistry Technical Leader and Crime Laboratory Quality Manager of the environmental control issue
- Measure the laboratory environmental conditions using a hygrometer and thermometer. ~~Laboratory environmental conditions shall be monitored by a Drug Chemist each working day using a hygrometer, thermometer and barometer.~~

7.2. Record the humidity, temperature and air pressure on the environmental log.

7.3. If the humidity exceeds 80% or the temperature is not in the range of 60 – 90 °F (15.6–32.2 °C), stop all analyses until environmental conditions return to acceptable levels.

7.3.1. All instruments shall be subjected to any post shutdown checks as described in the Drug Chemistry Unit Technical Procedures.

8. Safety – Refer to the CCBI Crime Laboratory Safety Manual

9. Millipore Elix Advantage Water Purification System with E-POD Unit

9.1. Prior to dispensing deionized water ensure that the resistivity displayed on the E-POD unit is greater than 1.0 MOhm.cm.

9.2. To dispense water press down on the E-POD Unit plunger. Push slightly for low flow. Push down and hold for high flow. Push completely down for continuous high flow and push down again to stop.

9.3. Maintenance

9.3.1. Replace the Progard Pack, Reservoir Vent Filter and~~,~~ UV 254 lamp ~~and POD Pak~~ when prompted by the System. Refer to 12.4.

9.3.2. Clean the Inlet Strainer and RO Cartridge when prompted by the System. Refer to 12.4

9.3.3. Record maintenance in a logbook maintained in the Drug Chemistry Unit.

9.4. Record System alarms in the logbook and notify the Drug Chemistry Unit Technical Leader if service is required.

10. Safety – Refer to the CCBI Crime Laboratory Safety Manual

11. Records

11.1. Microscope log
11.2. Refrigerator log
11.3. Environmental log

12. References

12.1. *Nikon Polarizing Microscope Eclipse E400Pol Instructions*, Nikon Inc, Melville, NY, M216E 98.8.VF.1.

12.2. *Nikon Stereoscopic Microscope SMZ645/SMZ660 Instructions*, Nikon Inc, Melville, NY, M219E 98.10.VH.1.

12.3. *Circular Fluorescent Microscope Illuminator Model 9 Operator Manual*, Stocker & Yale, Salem, NH, #09910009 Rev, A.

12.4. *Elix Advantage 3/5/10/15 System User Manual*, Millipore Corp, France, FTPF11337-V1.0, 02/2010.

Revision History		
Effective Date	Version Number	Reason
1/1/13	1	ISO Compliance
11/6/2015	2	Update Millipore maintenance in 5.4 and 9.3.1. Update environmental monitoring in 7.1.

4: Technical Procedure for Balances

1. **Purpose / Scope -** This procedure provides direction for the calibration and use of balances (scales) in the Drug Chemistry Unit of the Raleigh/Wake City-County Bureau of Identification

2. Definitions

2.1. Calibration - Checking or adjusting (by comparison with a standard) the accuracy of a measuring instrument.
2.2. Quality control check - Periodic confirmation of the reliability of equipment, instrumentation, and/or reagents.
2.3. Performance verification - The initial confirmation of the reliability of a previously or externally validated method or instrument.
2.4. Reference standard - Measurement standard designated for the calibration of other measurement standards (reference standards or equipment.)

3. Abbreviations

3.1. Refer to the Drug Chemistry Unit Technical Procedure for Drug Chemistry Analysis
3.2. Wt – weight
3.3. Rec'd – received
3.4. Ret'd – returned

4. Equipment and Materials

4.1. Equipment

4.1.1.A&D model HW-100KA1 digital platform scale
4.1.2. Mettler Toledo model XP205 balance equipped with antistatic unit
4.1.3. Mettler Toledo model XP6002S and XPE6002S balances
4.1.4. Laboratory environmental monitor – barometer, hygrometer, thermometer, NIST traceable

4.2. Materials

4.2.1. Paper, boxes, plastic bags, metal pans or other weighing vessels
4.2.2. Weights, non-NIST traceable: 100 mg, 1 g and 25 lb

4.3. Reference Standards

4.3.1.NIST traceable weights: 1 mg, 100 mg, 2 g, 100 g, 200 g, 1 kg, 5 kg, 20 kg

5. Standards and Controls

5.1. A balance logbook shall be maintained near each balance. The balance logbook shall contain the corresponding balance log and any manufacturer's certificates, performance verification documentation and maintenance documentation.

5.2. Leave balances powered on.

5.3. When the balance has been placed out of service (e.g., maintenance, malfunction, leaving direct control of the Laboratory), a Monthly Quality Control Check must be successfully performed prior to placing the instrument back in service, refer to section 7.

5.4. The Drug Chemist shall record any malfunctions or error messages in the balance log, notify the Drug Chemistry Technical Leader of any malfunctions or error messages and place the instrument out of service by marking the balance log "Out of Service."

5.5. The Drug Chemistry Technical Leader shall examine the effect(s), if any, of a malfunction or error message on analysis results and implement the CCBI Laboratory Procedure for Corrective and Preventive Action as required.

5.6. Reference Standard Weights and Weights

5.6.1. A reference standard weight logbook shall be maintained in the Drug Chemistry Unit. The reference standard logbook shall contain the calibration certificates demonstrating traceability to NIST.

5.6.2. Store reference standard weights and weights in their manufacturer supplied storage container, if available, or other container in the Drug Chemistry Unit. Keep the container securely closed when not in use.

5.6.3. Use gloves, tweezers and / or weight handles to handle reference standard weights and weights. Do not handle reference standard weights and weights with bare hands. Ensure that all surfaces that reference standard weights and weights may come in contact with are clean.

5.6.4. Reference standard weights shall be calibrated on an annual basis by an approved vendor that provides traceability to NIST and is accredited to ISO/IEC 17025:2005 by an accrediting body that is a signatory to the IAAC MultiLateral Recognition Arrangement or the ILAC Mutual Recognition Arrangement with a scope of accreditation covering the calibration performed.

5.6.4.1. When reference standard weights are transported outside of the laboratory for calibration they must be transported in their manufacturer supplied storage container, if available. If a manufacturer supplied storage container is not available, another container may be used. The container must be securely packaged to prevent damage.

5.6.4.2. If the reference standard weights must be shipped, they must be shipped by a carrier providing tracking information. Maintain the tracking information with the calibration documentation in the reference standard weight logbook.

5.6.4.3. Upon return to the laboratory the reference standard weight must be inspected for any damage. Clearly label any damaged reference standard weights "Out of Service" and notify the Drug Chemistry Technical Leader. Reference standard weights marked "Out of Service" shall not be used.

5.6.5. A list of reference standard weights, serial numbers and service/calibration due dates shall be maintained by the Drug Chemistry Unit Technical Leader on the shared drive in the Drug Chemistry Unit folder.

5.6.6. Certificates of calibration issued by the vendor must include the uncertainty and shall be maintained by the Drug Chemistry Unit Technical Leader in the Drug Chemistry Unit.

6. Calibrations

6.1. Calibration for all Drug Chemistry Unit balances, refer to 4.1, shall be performed on an annual basis by an approved vendor that provides traceability to NIST and is accredited to ISO/IEC 17025:2005 by an accrediting body that is a signatory to the IAAC MultiLateral Recognition Arrangement or the ILAC Mutual Recognition Arrangement with a scope of accreditation covering the calibration performed.

6.2. Balances shall be labeled with the calibration due date of the next calibration. A list of balances, serial numbers and service/calibration due dates shall be maintained by the Drug Chemistry Unit Technical Leader on the shared drive in the Drug Chemistry Unit folder.

6.3. Certificates of calibration issued by the vendor must include the uncertainty and shall be maintained in the balance logbook.

7. Monthly QCC

7.1. The Drug Chemist shall perform a monthly QCC on each balance using reference standard weight(s).

7.2. If the balance is not level, follow manufacturer's recommendations for leveling.

7.3. For the Mettler Toledo balances, press the internal adjustment key and allow the balance to complete the internal adjustment function.

7.4. Zero the balance with nothing on the pan.

7.5. Place a reference standard weight on the pan.

7.6. Record the actual weight displayed.

7.7. If results are within the acceptable range listed for the model, the balance may be used for casework.

Mettler Toledo model # XP6002S and XPE6002S

Reference Standard Certified Weight	**Acceptable Range** (± 0.04 gram)
~~5000.0161 and 1000.00045~~ 5000.0097 and 1000.00061 gram	~~5999.98 – 6000.06~~ 5999.97 – 6000.05 g
~~5000.0161~~ 5000.0097 gram	~~4999.98 – 5000.06~~ 4999.97 – 5000.05g
~~1000.00045~~ 1000.00061 gram	999.96 – 1000.04 g
	Acceptable Range (± 0.02 gram)
~~200.000219~~ 200.000209 gram	199.98 – 200.02 g
~~100.000028~~ 100.000021 gram	99.98 - 100.02 g
~~2.0000132~~ 2.0000129 gram	1.98 - 2.02 g
~~0.0999970~~ 0.0999968 gram	0.08 - 0.12 g

Mettler Toledo model # XP205

Reference Standard Certified Weight	**Acceptable Range** (± certified weight tolerance)
~~200.000219~~ 200.000209 gram	~~199.99622 – 200.00422~~ 199.99621 – 200.00421 g
~~100.000028~~ 100.000021 gram	~~99.99978 – 100.00028~~ 99.99977 – 100.00027g
	Acceptable Range (± three times repeatability (s) of the balance)
~~2.0000132~~ 2.0000129 gram	1.99992 – 2.00010 g
~~0.0999970~~ 0.0999968 gram	0.09991 - 0.10009 g
SN: B309079044: ~~0.0010045~~ 0.0010037 gram or SN: B403234453: ~~0.0010032~~ 0.0010034 gram	0.00091 – 0.00109 g

A&D model #HW-100KA1

Reference Standard Certified Weight	**Acceptable Range** (± two times repeatability (s) of the balance)
~~5000.0161~~ 5000.0097 gram (11.02 lb)	10.98 – 11.06 lb
~~20000.012~~ 20000.017 gram (44.09 lb)	44.05 – 44.13 lb

7.8. If the results are outside these parameters, the balance shall not be used until all necessary steps have been taken to bring the balance into compliance.

7.8.1. Steps may include cleaning, leveling, zeroing and performing the internal adjustment. If the problem cannot be corrected, clearly mark the balance logbook "Out of Service" and notify the Drug Chemistry Technical Leader. Service by an approved service contractor may need to be scheduled.

7.9. Record the results of the QCC and any action taken in the Balance Logbook.

8. Daily QCC

8.1. The Drug Chemist shall perform a daily QCC on each balance using a reference standard weight prior to use for casework each day.

8.2. Follow the procedure in 7. Use a 2 gram and a 5 kg weight for the Mettler Toledo XP6002S and XPE6002S. Use only the 20 kg weight for the AND HW-100KA1. Use only the 1 mg weight for the XP205.

9. Process Measurement Assurance

9.1. Check standard surrogates shall be maintained in the Drug Chemistry Unit for ongoing data collection on the weight determination process. The surrogates shall consist of a heat sealed plastic bag containing approximately the amount of material indicated.

9.2. Measure and record the weight of the check standard surrogates each week that weight measurements are conducted in the Drug Chemistry Unit.

9.3. One day each week measure and record the temperature, humidity and air pressure at the balance (scale) location using a laboratory environmental monitor. Measure and record the weight of each applicable check standard surrogate in the morning and in the afternoon.

9.3.1. Each week the measurements should be made on the next day of the week so that the measurements rotate through the days of the work week.

9.3.2. Each active Drug Chemist must participate in the data collection. Data collection should rotate so that each active Drug Chemist participates equally.

9.3.3. Balances that are used exclusively by an individual Drug Chemist need only have data collected by that individual.

9.4. Perform the measurements using tare vessels, as in casework, i.e., when appropriate remove the tare vessel from the balance, place the check standard on the tare vessel and return the tare vessel and check standard to the balance together. Tare vessels and check standard surrogates are selected to mimic casework as closely as possible:

AND HW100KA1 scale:
Tare vessel: Cardboard box lined with a plastic bag
Check standard surrogate: Heat sealed plastic bag containing approximately 5 kg of powder material.

Mettler XP205:
Tare vessel: Use weighing paper, 3" X 3"
Check standard surrogate: Heat sealed plastic bag containing approximately 0.05 g of powder material.

Mettler XP6002S and XPE6002S:
Tare vessel: Rectangular metal pan
Check standard surrogate: Heat sealed plastic bag containing approximately 1 kg of powder material.

Tare vessel: approximately one-half sheet of 8.5" X 11" paper
Check standard surrogate: Heat sealed plastic bag containing approximately 1 g of powder material.

9.5. Record the measurements on the corresponding balance measurement assurance log.

9.6. Enter the measurements on the corresponding balance measurement assurance control chart located on the shared drive.

9.6.1. Evaluate the measurement. If the measurement result:

9.6.1.1. falls outside of the range of ± three standard deviations, or

9.6.1.2. two out of three consecutive measurements are on the same side of the centerline and fall outside of the range of ± two standard deviations, or

9.6.1.3. four out of five consecutive measurements are on the same side of the centerline and fall outside of the range of ± one standard deviation, or

9.6.1.4. eight consecutive measurements are on the same side of the centerline, or 10 out of 11, or 12 out of 14, or 16 out of 20, or

9.6.1.5. there is an obvious consistent or persistent pattern that suggests something unusual about the process, notify the Drug Chemistry Unit Technical Leader for evaluation of the process.

10. Procedure

10.1. Record weights on the XP6002S and XPE6002S balances in grams to two decimal places. Record weights on the XP205 balance in grams to five decimal places. Record weights on the HW100KA1 balance in pounds to two decimal places. Refer to 11.1 for conversion of units.

10.1.1. For net weights of analyzed material use each balance only at or above the following minimum weights:

Balance	Minimum weight
XP205	N/A, refer to 12.1.2
XP6002S and XPE6002S	N/A, refer to 12.1.2
AND HWK100KA1	1.00 lb (0.45 kg)

10.1.2. Use each balance only at or below the following maximum weights:

Balance	Maximum Weight
XP205	200 g
XP6002S and XPE6002S	6000 g
AND HWK100KA1	220.46 lb (100 kg)

10.1.3. For net weights of material use the XP205 balance only at or below 2.00 grams.

10.2. Ensure that the balance is level, refer to 7.3. Zero the balance and ensure that the balance displays zero to the appropriate number of decimal places, refer to 10.1.

10.3. Place the weighing vessel on the balance and allow the reading to stabilize. For the XP205 allow at least 10 seconds.

10.4. Remove evidence from packaging material, if possible, and place in/on the tared vessel. If packaging is not removed record the weight as a gross weight on the appropriate Drug Chemistry Worksheet. If packaging is removed record the weight as a net weight on the appropriate Drug Chemistry Worksheet.

10.5. Record all digits displayed by the balance (to two decimal places for the XP6002S and the A&D scale, to five decimal places for the XP205 balance) on the appropriate Drug Chemistry Worksheet, refer to the Drug Chemistry Unit Technical Procedure for Drug Chemistry Analysis.

10.6. For the weight of the material to be returned, either replace the weighing vessel back on the undisturbed balance without taring or tare a new weighing vessel and transfer the evidence to the tared vessel.

10.7. Record all digits displayed by the balance on the appropriate Drug Chemistry Worksheet.

11. Calculations

11.1. When conversion of units is needed, the following NIST Conversion factor shall be used and the result rounded as directed in 16.10:

1 pound = 0.45359237 kilograms

12. Reporting

12.1. Report the measured weight, as recorded, along with the uncertainty of measurement, refer to the Drug Chemistry Unit Technical Procedure for Uncertainty of Measurement.

12.1.1. When the measured weight to be reported was measured to two decimal places and is less than 0.20 gram report the weight as *"less than 0.2 gram.*"

12.1.2. When the measured weight to be reported was measured to five decimal places and is less than 0.001 g report the weight as "*less than 0.001 gram*."

12.2. When the numerical value is a weight associated with unanalyzed material the EU need not be included on the report.

12.3. When the weight to be reported is a sum of measured weights, sum the uncertainty of measurement associated with each measured weight to determine the uncertainty of measurement to be included on the report.

12.4. For estimated total weights determined with hypergeometric sampling, refer to the Drug Chemistry Analysis Technical Procedure.

13. Limitations

13.1. When the reference standard weights are unavailable for the Daily QCC due to annual calibration, other non-reference standard weights may be used. For the XP6002S and XPE6002S use a 1 gram weight. For the XP205 use a 100 mg (0.1 g) weight. For the A&D scale use a 25 pound weight. Each weight must be weighed on the applicable balance prior to the reference standard weights becoming unavailable and be within the applicable following acceptable range. Each Daily QCC must be within the following acceptable range. Record the manufacturer and serial number of each weight used in the balance logbook.

Mettler Toledo model # XP6002S and XPE6002S

Weight	Acceptable Range
1 gram	0.98 - 1.02 g

Mettler Toledo model # XP205

Weight	Acceptable Range
0.1 gram	0.09991 - 0.10009 g

A&D model #HW-100KA1

Weight	Acceptable Range
25 lb	24.93 – 25.07 lb

14. Safety – Refer to the CCBI Crime Laboratory Safety Manual

15. Records

15.1. Balance log AND
15.2. Balance log Mettler XP205
15.3. Balance log Mettler XP6002S and XPE6002S
15.4. Measurement assurance log AND
15.5. Measurement assurance log Mettler XP205
15.6. Measurement assurance log Mettler XP6002S andXPE6002S

16. References

16.1. *Mettler Toledo B Balance Line Operating Instruction***s**, Mettler-Toledo, Switzerland, P11780194**.**

16.2. *HV/HW Series Instruction Manual***,** A&D Engineering Inc., Milpitas, CA, V.1.C-95.04.03.

16.3. Butcher, K.S, et al., ed. *The International System of Units (SI) – Conversion Factors for General Use*. National Institute of Standards and Technology, NIST Special Publication: U.S Department of Commerce, May 2006: 11.

16.4. *Mettler Toledo Excellence Plus Analytical Balances XP Models – Part 1*, 11781066, Mettler-Toledo, Switzerland, May 2012.

16.5. *Mettler Toledo Excellence Plus Precision Balances XP Models – Part 1*, 11781055, Mettler-Toledo, Switzerland, May 2012.

16.6. *Mettler Toledo Excellence Plus Balances XP Models – Part 2*, 11781077, Mettler-Toledo, Switzerland, October, 2010.

16.7. *Mettler Toledo Excellence Plus Balances XP Models – Part 3*, 11781338, Mettler-Toledo, Switzerland, October, 2010.

16.8. *Mettler Toledo Discharging Power Pack Operating Instructions,* Haug GmbH & Co. KG, D-032 V02, March 29, 2012.

16.9. *One Point Ionizer Operating Instructions,* Haug GmbH & Co. KG, D-0265, December 22, 2004.

16.10. *Guide for the Use of the International System of Units (SI).* NIST Special Publication 811, 2008 Ed., (March 2008; 2nd printing November 2008). pp. 43-44, 53.

16.11. *ASTM Standard E2587-12. "Standard Practice for Use of Control Charts in Statistical Process Control."* ASTM International: West Conshohocken, PA, 2009, www.astm.org.

16.12. *ASCLD/LAB Guidance on Measurement Traceability – Measurement Assurance,* ASCLD/LAB, AL-PD-3059 Ver 1.0.

Revision History		
Effective Date	Version Number	Reason
1/1/13	1	ISO Compliance
3/27/13	2	Reference standard weight modification
8/7/13	3	Incorporation of Uncertainty of Measurement and Measurement Assurance
9/16/13	4	Change to acceptable range
2/19/14	5	Removal of Mettler PB3002
4/25/14	6	Addition of 1kg and 1mg reference standard weights
11/7/14	7	Update QCC acceptable range and add additional daily QCC weight for XP6002S balances
2/16/15	8	Added model XPE6002S balance and corrected reference in table in 10.1.1.
2/25/15	9	Added reference to 1 kg weight in 4.3.1 and updated certified weight values and acceptable ranges in 7.7
11/6/15	10	Added 5 kg weight conversion in 7.7 and updated minimum weight to be reported in 12.1 to 0.2 gram.
2/25/16	11	Updated certified weight values and acceptable ranges in 7.7

5: Technical Procedures 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 Raleigh/Wake City-County Bureau of Identification Crime 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. **Abbreviations**

   3.1. Refer to the Drug Chemistry Unit Technical Procedure for Drug Chemistry Analysis
   3.2. UOM – uncertainty of measurement
   3.3. CU – combined uncertainty
   3.4. EU – expanded uncertainty

4. **Procedure**

   4.1. The Drug Chemistry Technical Leader shall determine an estimation of the UOM for each test method for which a numerical value is reported on a laboratory report. The specific measuring device or instrument used for a reported test result must be evaluated in the estimation of the UOM for that test method.

   4.2. When established, 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.

   4.3. Laboratory environmental conditions shall be monitored and any additional effect on UOM shall be evaluated upon collection of data. Refer to the Drug Chemistry Unit Technical Procedure for General Laboratory Equipment.

   4.4. 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.)

4.5. 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

4.6. 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.

4.7. 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.

4.8. 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.

4.9. 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. When conversion of units is necessary, perform the conversion after any summing and use the appropriate NIST conversion factor with the result rounded up: 1 pound = 0.45359237 kilograms. When the numerical value is a gross weight or a weight associated with unanalyzed material the EU need not be included on the report.

4.10. 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 of the material: 4.00 grams ± 0.12 gram at a coverage probability of 99.5%.
Net weight of the material: 0.01875 gram ± 0.00053 gram at a coverage probability of 99.5%.
Net weight of the material: 10.27 pounds ± 0.07 pound at a coverage probability of 99.5%.

4.10.1. When compliance with a statutory limit is in question, such as trafficking limits and other weight thresholds listed in the North Carolina Controlled Substances Act, add an additional statement to the report to clearly communicate this information. When notified that a submission is intended for prosecution in Federal courts, refer to the United States Sentencing Commission Guidelines Manual for applicable weight thresholds.

Example:

Taking the estimated measurement uncertainty into consideration, there is a possibility that the net weight of the material is less than 28 grams.

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

- Define the measurand
- State how traceability is established for the measurement
- State the equipment (measuring device(s)) used
- State all uncertainty components considered
- Identify all uncertainty components of significance and how they were evaluated
- Contain the data used to estimate repeatability and / or reproducibility
- Contain all calculations performed
- State the combined standard uncertainty, CU, the coverage factor, k, the coverage probability, C, and the resulting expanded uncertainty, EU
- The minimum due date for the review/recalculation of the measurement uncertainty, refer to 4.2.

5. Calculations

5.1. $CU = \sqrt{(u_1^2 + u_2^2 + u_3^2 \ldots)}$

5.2. $EU = CU * k$

6. References

6.1. *ASCLD/LAB Level 100A Traceability presentation*, Copyright 2011; Heusser Neweigh, LLC & ASCLD/LAB.

6.2. *ASCLD/LAB Level 100B Measurement Assurance presentation*, Copyright 2011; Heusser Neweigh, LLC & ASCLD/LAB.

6.3. *ASCLD/LAB Level 100C Measurement Uncertainty Concepts presentation*, Copyright 2011; Heusser Neweigh, LLC & ASCLD/LAB.

6.4. *ASCLD/LAB Level 200 Measurement Confidence for the Forensic Laboratory: Measurement Uncertainty in Drug Chemistry presentation*, Copyright 2011; Heusser Neweigh, LLC & ASCLD/LAB.

6.5. *ASCLD/LAB Level 200 Measurement Confidence for the Forensic Laboratory: Measurement Uncertainty in Toxicology Testing presentation***,** Copyright 2011; Heusser Neweigh, LLC & ASCLD/LAB.

6.6. *Introduction to Measurement Uncertainty course***,** LeBeau, Marc A., Ph.D., 2009, RTI International

6.7. *Introduction to Measurement Uncertainty – Practical Examples Part II course***,** LeBeau, Marc A., Ph.D., 2010, RTI International

6.8. *Introduction to Measurement Uncertainty – Practical Examples Part III course***,** LeBeau, Marc A., Ph.D., 2010, RTI International

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)

6.10. *"Supplemental Document SD-3, Part IVC - Measurement Uncertainty for Weight Determinations in Seized Drug Analysis."* Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG). July 7, 2011.

6.11. *ASCLD/LAB Policy on Measurement Uncertainty*, ASCLD/LAB, AL-PD-3060 Ver 1.1.

6.12. *ASCLD/LAB Guidance on the Estimation of Measurement Uncertainty – Overview*, ASCLD/LAB, AL-PD-3061 Ver 1.0.

6.13. *ASCLD/LAB Guidance on the Estimation of Measurement Uncertainty – ANNEX A, Details on the NIST 8-Step Process*, ASCLD/LAB, AL-PD-3062 Ver 1.0.

6.14. *ASCLD/LAB Guidance on the Estimation of Measurement Uncertainty – ANNEX B, Drug Chemistry Discipline Three Examples – Weight, Volume and Purity Determination*, ASCLD/LAB, AL-PD-3063 Ver 1.0.

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.

Revision History		
Effective Date	Version Number	Reason
1/1/13	1	ISO Compliance
8/7/13	2	Incorporation or Uncertainty of Measurement and Measurement Assurance
2/16/15	3	Added lines 4.10.1, 5.2, 6.15. Addition to line 4.9.

6: Technical Procedure for Quality Assurance

1. **Purpose / Scope** – This procedure provides direction for the receipt and quality assurances of laboratory supplies, equipment, reagents, reference collections, reference standards and reference materials that affect casework in the Drug Chemistry Unit of the Raleigh/Wake City-County Bureau of Identification Crime Laboratory

2. **Definitions**

2.1. Quality control check – Periodic confirmation of the reliability of equipment, instrumentation, and/or reagents.
2.2. Performance verification – The initial confirmation of the reliability of a previously or externally validated method or instrument.
2.3. Commercial reagent - A purchased solvent or chemical.
2.4. Critical reagent - Chemicals or reagents which critically affect the quality of tests which do not have their reliability verified as part of the quality control checks in a Drug Chemistry Unit Technical Procedure.
2.5. Prepared reagent - Mixture of two or more reagents or a dilution.
2.6. Reference standard - Measurement standard designated for the calibration of other measurement standards (reference standards or equipment.)
2.7. Reference material - Material sufficiently homogeneous and stable with reference to specified properties, which has been established to be fit for its intended use in measurement or in examination of nominal properties.
2.8. Primary reference material - Any reference material obtained from a commercial source which has documentation issued by the manufacturer certifying its chemical composition or has documentation stating the manufacturer's specifications for the material. This material may be certified reference material if available and practicable.
2.9. Secondary reference material - Reference material from a non-commercial source or from a commercial source which does not have authenticating documentation from the manufacturer or is derived from reference material.
2.10. Authenticating documentation - A certificate of analysis provided by the manufacturer certifying chemical composition or a statement of the manufacturer's specifications or any published spectral data from an informed treatise generally accepted in the field that identifies a chemical substance.
2.11. Purchasing documentation – Any requisition forms, vendor quotes and packing slips associated with the purchase and receipt of Drug Chemistry Unit laboratory supplies, equipment, reagents, reference collections, reference standards and reference materials.
2.12. Stock Container – A container of reagent prepared to serve as a reserve source of the reagent from which Use Containers are prepared. Stock containers shall not be used directly for analysis.
2.13. Use Container – A container of reagent used directly for analysis.
2.14. Training standards – Controlled substances and non-controlled substances used solely for training purposes.

3. Abbreviations

3.1. Refer to the Drug Chemistry Unit Technical Procedure for Drug Chemistry Analysis

4. Procedure for Laboratory Supplies and Commercial Reagents

4.1. Prior to use, received laboratory supplies and commercial reagents shall be inspected for compliance with specifications as stated in the purchasing documentation. The purchasing documentation shall reflect any specifications required in the applicable Drug Chemistry Unit Technical Procedure.

4.2. When laboratory supplies are found to meet specifications they shall be stored for use in the Drug Chemistry Unit. When commercial reagents are found to meet specifications they shall be marked with the initials of the receiving Drug Chemist and the date received. Update the Drug Chemistry Unit Chemical Inventory log maintained in the Drug Chemistry Unit folder on the shared drive when the material is received and when it is emptied / disposed. Commercial reagents shall be stored in accordance with the manufacturer's specifications, if applicable, and in accordance with the CCBI Crime Laboratory Safety Manual.

4.2.1. If applicable, a copy of the packing slip shall be marked with the initials of the receiving Drug Chemist and the date of receipt.

4.2.2. The requisition shall be marked with the date of receipt and the initials of the receiving Drug Chemist.

4.2.3. Forward the original completed requisition to the Deputy Director and maintain a copy in the Drug Chemistry Unit along with any other associated purchasing documentation.

4.3. Laboratory supplies and commercial reagents that do not meet specifications shall be not be used. They shall be stored in a separate area and clearly marked "Not for Use" until they can be returned or otherwise disposed.

4.3.1. Mark the packing slip, if applicable, and the requisition form with the discrepancy, initials of the receiving Drug Chemist and forward to the Forensic Quality Manager and Deputy Director. Maintain a copy in the Drug Chemistry Unit along with any other associated purchasing documentation.

4.4. Upon being opened, commercial reagent containers shall be marked as opened along with the initials of the Drug Chemist and the date.

4.4.1. When a commercial reagent is transferred to another container it shall be labeled with the following:

4.4.1.1. Identity and grade, if applicable

4.4.1.2. Supplier and lot number
4.4.1.3. Initials of the Drug Chemist
4.4.1.4. Date
4.4.1.5. Expiration date, if applicable.

5. Procedure for Prepared Reagents

5.1. Reagents may be prepared in any amount provided that the component ratios in the Drug Chemistry Unit Technical Procedure are kept constant.

5.2. A stock container is a container of reagent prepared to serve as a reserve source of the reagent from which use containers are prepared. Stock containers shall not be used directly for analysis.

5.3. A use container is a container of reagent used directly for analysis.

5.4. Labeling

5.4.1. Lot numbers for stock containers and use containers of prepared reagents shall be assigned using lot number designations as specified in the Drug Chemistry Unit Technical Procedure.

5.4.2. Stock containers of prepared reagents shall be labeled "Stock – Not for Direct Use."

5.4.3. Stock containers of prepared reagents shall be labeled with the following:

5.4.3.1.1. Identity of the reagent
5.4.3.1.2. Initials of preparer
5.4.3.1.3. Date of preparation
5.4.3.1.4. Lot number
5.4.3.1.5. Expiration date

5.4.4. Use containers of prepared reagents shall be labeled with the following:

5.4.4.1.1. Identity of the reagent
5.4.4.1.2. Initials of preparer
5.4.4.1.3. Date of preparation
5.4.4.1.4. Lot number
5.4.4.1.5. Expiration date
5.4.4.1.6. QCC due date

5.4.5. Each new container of prepared reagent shall be documented in the reagent log with the following:

5.4.5.1.1. Identity of the reagent and lot number
5.4.5.1.2. Reference to the Drug Chemistry Unit Technical Procedure followed for preparation

5.4.5.1.3. Initials of preparer
5.4.5.1.4. Date of preparation
5.4.5.1.5. Expiration date
5.4.5.1.6. QCC result and supplier and lot number of any reference material used
5.4.5.1.7. Component(s) and supplier and lot number

5.5. Storage

5.5.1. Reagents shall be stored in closed containers.

5.5.2. All stock containers shall be stored in the chemical storage refrigerator of the Drug Chemistry Unit unless otherwise specified in the Drug Chemistry Unit Technical Procedure.

5.5.3. All use containers shall be stored on the countertop or under the hood, unless otherwise specified in the Drug Chemistry Unit Technical Procedure.

5.6. Expiration Dates

5.6.1. Stock Containers have a three year expiry unless otherwise specified in the Drug Chemistry Unit Technical Procedure.

5.6.2. Use containers have a one year expiry date unless otherwise specified in the Drug Chemistry Unit Technical Procedure.

5.6.3. Use Containers with an expiry greater than six months must be have QCC(s) repeated every six months to ensure reagent reliability.

5.7. Quality Control Checks

5.7.1. Prepared reagents shall be quality control checked according to the Drug Chemistry Unit Technical Procedure prior to initial use and use containers with an expiry of greater than six months shall have QCC(s) repeated every six months to ensure reagent reliability.

5.7.2. Document quality control checks in the reagent log with the following:

5.7.2.1. Date performed
5.7.2.2. Initials of Drug Chemist
5.7.2.3. Reference material, supplier and lot number
5.7.2.4. QCC result
5.7.2.5. Due date for next QCC

5.7.3. The next QCC due date shall be listed on the use container, if applicable.

6. Procedure for Reference Materials

6.1. Reference material containers shall be received and labeled by the Drug Chemist as directed for commercial reagents, refer to Section 4. Store Reference Materials that have not been approved for use in a location that is clearly labeled "Pending Approval." Update the Drug Chemistry Unit Reference Material Inventory log maintained in the Drug Chemistry Unit folder on the shared drive when the material is received and when it is emptied / disposed.

6.1.1. In the event that the container is too small to be labeled as required, the reference material may be stored in a larger container or the required information may be recorded in the reference material log. Drug Chemists shall refer to the reference material log for any information not recorded on the container prior to using reference material.

6.1.2. If material is to be obtained from a submission for use as a reference material a drug acquisition form must be completed by the Drug Chemist and approved by the Drug Chemistry Technical Leader. The Drug Chemist must record the amount removed and the purpose on the Drug Chemistry worksheet for inclusion in the case file. The Drug Chemistry Technical Leader must verify, with initials and date, this entry in the case file. The drug acquisition form shall be maintained in the Drug Chemistry Unit with any data generated and any authenticating documentation.

6.2. Reference materials used in the Drug Chemistry Unit for the identification of controlled substances and in quality control checks are critical reagents.

6.2.1. Prior to use, a Drug Chemist shall analyze primary reference materials by infrared and/or mass spectrometry, at a minimum, using Drug Chemistry Unit Technical Procedures to ensure that the materials are appropriately identified and suitable for use.

6.2.1.1. The Technical Leader shall qualitatively evaluate the data produced. The primary reference material must be found to be substantially comparable to authenticating documentation, reference material, and/or published spectral libraries. Reference material that is not found to be substantially comparable to authenticating documentation, reference material and/or published spectral libraries shall not be used.

6.2.1.2. Primary reference material found to be suitable for use shall be marked "APD" along with the date, Technical Leader initials and the QCC due date, see 6.2.1.3. All data and authenticating documentation shall be marked by the Drug Chemist with initials and date and maintained in the Drug Chemistry Unit. Upon approval the Technical Leader shall move the Reference Material to a location that is clearly labeled "Approved" and update the appropriate in-house reference collection.

6.2.1.3. The primary reference material evaluation must be repeated after one year if the reference material is to be used for identification of a controlled substance or in a QCC.

6.2.2. Prior to use, a Drug Chemist shall analyze secondary reference materials by infrared and/or mass spectrometry, at a minimum, using Drug Chemistry Unit Technical Procedures to ensure that they are appropriately identified and suitable for use.

6.2.2.1. Secondary reference materials must be approved by the Drug Chemistry Technical Leader prior to use.

6.2.2.2. Secondary reference material found to be suitable for use shall be marked "APD" along with the date, Drug Chemistry Technical Leader initials and the QCC due date, see 6.2.2.3. All data and documentation shall be marked by the analyzing Drug Chemist with initials and date and be maintained in the Drug Chemistry Unit. Upon approval the Technical Leader shall move the Reference Material to a location that is clearly labeled "Approved" and update the appropriate in-house reference collection.

6.2.2.3. The secondary reference material evaluation must be repeated after one year if the reference material is to be used for identification of a controlled substance or in a QCC.

6.3. Reference materials shall be maintained in room C1401 and stored according to the manufacturer's instructions, if applicable.

6.4. A reference material log shall be maintained.

6.4.1. Each Drug Chemist who uses reference materials shall update the reference material log with initials and date. For solid material include the gross weight of reference material prior to removing material for use, the amount removed and the gross weight of reference material as returned to storage. For dilute liquids purchased in individual quantities of 1.5 ml or less, include only the volume removed.

6.5. An audit of the Drug Chemistry reference materials shall be conducted annually according to the CCBI Administrative Procedure for Annual Quality Audits.

7. Procedure for Training Standards

7.1. Training standards shall be marked as such and maintained separately from reference materials. Training standards shall be maintained in room C1401 and stored according to the manufacturer's instructions, if applicable

7.2. Documentation demonstrating the identity of training standards shall be maintained in the Drug Chemistry Unit.

7.3. All training standards shall be labeled with a unique identifier.

7.4. The Drug Chemistry Technical Leader shall maintain an inventory and log of training standard usage.

7.5. An audit of the Drug Chemistry training standards shall be conducted annually according to the CCBI Administrative Procedure for Annual Quality Audits.

8. Procedure for In-house Generated Reference Collections

8.1. Spectral reference collections generated within the Laboratory will be traceable to primary reference materials, if practicable, otherwise secondary reference materials may be used.

8.2. Current and archived in-house generated spectral reference collections shall be maintained by the Drug Chemistry Technical Leader.

8.3. Spectral reference collections will be titled with:

8.3.1.1.1. Technique identifier (i.e., MS, IR,).
8.3.1.1.2. "ccbi"
8.3.1.1.3. Date in the format YYYYMMDD. Example "MSccbi20101207"

9. Procedure for Reference Standards

9.1. Refer to the Drug Chemistry Unit Technical Procedure for Balances

10. Safety – Refer to the CCBI Crime Laboratory Safety Manual

11. Records

11.1. Reagent log
11.2. Drug acquisition form
11.3. Reference material log

12. References

12.1. *ASCLD/LAB Policy on Measurement Traceability,* ASCLD/LAB, AL-PD-3057 Ver 1.1.

12.2. *ASCLD/LAB Guidance on Measurement Traceability,* ASCLD/LAB, AL-PD-3058 Ver 1.0.

Revision History		
Effective Date	Version Number	Reason
1/1/13	1	ISO Compliance
8/7/13	2	Incorporation of Uncertainty of Measurement and Mesaurement Assurance
2/2/14	3	Changes to reference material storage
2/16/15	4	Additions to lines 4.2 and 6.1. Added reference material log to section 11.

7: Technical Procedures for Color Tests Technical Procedure

1. **Purpose / Scope** - This procedure provides direction for the preparation and use of color test in the Drug Chemistry Unit of the Raleigh/Wake City-County Bureau of Identification Crime Laboratory.

2. Definitions

2.1. Prepared reagent – Mixture of two or more reagents or a dilution.
2.2. Commercial reagent – A purchased solvent or chemical.
2.3. Performance verification – The initial confirmation of the reliability of a previously or externally validated method or instrument.
2.4. Quality control check – Periodic confirmation of the reliability of equipment, instrumentation, and/or reagents.
2.5. Reference material – Material sufficiently homogenous and stable with reference to specified properties, which has been established to be fit for its intended use in measurement or in examination of nominal properties.
2.6. Stock Container – a container of reagent prepared to serve as a reserve source of the reagent from which Use Containers are prepared. Stock containers shall not be used directly for analysis.
2.7. Use Container – a container of reagent used directly for analysis.

3. Abbreviations

3.1. Refer to the Drug Chemistry Unit Technical Procedure for Drug Chemistry Analysis
3.2. SNP - Simon's Test (Modified Sodium Nitroprusside)
3.3. *p*DMAB - *para*-Dimethylaminobenzaldehyde

4. Equipment, Materials and Reagents

4.1. Equipment

4.1.1. Balance

4.2. Materials

4.2.1. Beakers or other glass vessels
4.2.2. Test tubes
4.2.3. Funnel
4.2.4. Glass stirring rod
4.2.5. Graduated cylinder, class A
4.2.6. Pipettes with bulb
4.2.7. Porcelain spot plates

4.2.8. Reagent bottles and stock bottles, including amber
4.2.9. Spatula
4.2.10. Weigh boats or other weigh vessels
4.2.11. Filter paper
4.2.12. Scissors
4.2.13. Deionized water

4.3. Commercial Reagents

4.3.1. Sulfuric acid, ACS
4.3.2. Formaldehyde, approximately 40% aqueous, ACS
4.3.3. Vanillin, NF
4.3.4. Acetaldehyde, 99.5%
4.3.5. Ethanol, ACS
4.3.6. Chloroform, Optima
4.3.7. Cobalt (II) Thiocyanate
4.3.8. Ferric Chloride, anhydrous
4.3.9. Colbalt(II) Acetate, tetrahydrate
4.3.10. Methanol, Optima or GC Resolv
4.3.11. Isopropylamine, 99%
4.3.12. *Para*-Dimethylaminobenzaldehyde, ACS
4.3.13. Hydrochloric acid, ACS
4.3.14. Molybdic acid, ACS or Sodium Molybdate, dihydrate, ACS
4.3.15. Selenious acid, 98%
4.3.16. Copper(II) Sulfate, pentahydrate, ACS
4.3.17. Pyridine, ACS
4.3.18. Sodium Nitroprusside, dihydrate, ACS
4.3.19. Sodium Carbonate, anhydrous, ACS
4.3.20. Cobalt (II) Nitrate, hexahydrate, ACS
4.3.21. Glacial acetic acid, ACS grade

4.4. Reference materials

4.4.1. Heroin hydrochloride
4.4.2. Marijuana or *delta*-9-Tetrahydrocannabinol
4.4.3. Cocaine hydrochloride
4.4.4. Acetaminophen
4.4.5. Phenobarbital
4.4.6. Lysergic acid diethylamide
4.4.7. *Gamma*-hydroxybutyric acid
4.4.8. Methamphetamine

5. Standards and Controls

5.1. Reagents shall be prepared, labeled and stored in accordance with the Drug Chemistry Unit Technical Procedure for Receipt and Quality Assurance of Laboratory Supplies, Reagents, Reference Collections, Reference Standards and Reference Materials.

5.2. Perform positive and negative quality control checks on all Use Containers of color test reagents prior to use for analysis. The quality control checks must have acceptable results prior to use of the reagent for analysis. Refer to the CCBI Crime Laboratory Administrative Procedure for Corrective and Preventive Action if necessary.

5.3. Perform negative quality control checks (NQCC) according to the procedure listed for each color test with no sample present.

5.3.1. Acceptable result is no significant color formation, i.e., Negative.

5.3.2. If a significant color develops, take steps to ensure that the spot well is clean or use a new spot well or use a new culture tube.

5.3.3. If the significant color formation persists, dispose of the reagent and prepare a new lot of reagent.

5.3.4. Record any observations and the results of each quality control check in the Reagent Logbook.

5.4. Perform positive quality control checks (PQCC) according to the procedure listed for each color test using the specified reference material.

5.4.1. Refer to each color test for acceptable results.

5.4.1.1. If acceptable results are not observed, take steps to ensure that the spot well is clean or use a new spot well or use a new culture tube and repeat the PQCC. If the problem persists, dispose of the reagent and prepare a new lot of reagent.

5.4.2. Record any observations, the reference material identification and the results of each quality control check in the Reagent Logbook.

6. Color Tests

6.1. Marquis

6.1.1. Useful for general screening mostly with opium alkaloids, opioids and amphetamines.

6.1.2. Selected Characteristic Results:

Opiates, Opioids (morphine, heroin, oxycodone) - purple
Guaifenesin - purple

Methamphetamine - orange/brown
MDA/MDMA - purple/black
Aspirin - slow cherry red
Bufotenine and psilocin - green-brown

6.1.3. Preparation: Prepare a 1 % (v/v) solution of approximately 40% aqueous formaldehyde solution in concentrated sulfuric acid.

6.1.3.1. Storage: Amber glass.

6.1.3.2. Expiration: Stock container: One month
Use container: One month.

6.1.3.3. Lot number: Eight digit format year/month/day/Mq/initials of preparer.
Example: 20120131MqXXX

6.1.3.4. PQCC

6.1.3.4.1. Reference material: Heroin HCl
6.1.3.4.2. Acceptable result: Purple color observed

6.1.4. Procedure:

6.1.4.1. Add 1-2 drops of the reagent to a clean spot well or a new test tube and observe any reaction or color produced.

6.1.4.1.1. If a significant color develops, take steps to ensure that the spot well is clean or use a new spot well or use a new test tube.
6.1.4.1.2. If the significant color formation persists prepare a new lot of reagent.

6.1.4.2. Add a small amount of sample to the reagent.

6.1.4.3. Observe any reaction or color produced.

6.1.4.4. Record observations.

6.2. Duquenois-Levine (Modified)

6.2.1. Reacts with marijuana, hashish, and cannabinoids to produce a violet blue color that transfers to the chloroform layer.

6.2.2. Preparation: Dissolve 2.0 grams of vanillin and 2.5 milliliters of acetaldehyde in 100 milliliters of ethanol.

6.2.2.1. Storage: Amber glass.

6.2.2.2. Expiration: Stock container: Three years
Use container: Three months

6.2.2.3. Lot number: Eight digit format year/month/day/Duq/initials of preparer**.**
Example: 20120131DuqXXX

6.2.2.4. PQCC:

6.2.2.4.1. Reference material: Marijuana or Δ^9-Tetrahydrocannabinol.

6.2.2.4.2. Acceptable result: A violet blue color observed after the addition of acid and the violet color transfers to the chloroform layer, i.e., Positive.

6.2.3. Procedure

6.2.3.1. Place a small amount of sample in a culture tube or spot plate.

6.2.3.1.1. An evaporated petroleum ether or chloroform extract may be used. Record the preparation of the sample on the appropriate Drug Chemistry Worksheet form.

6.2.3.2. Add at least three drops of the Duquenois reagent prepared in 6.2.2. and mix thoroughly.

6.2.3.2.1. The liquid may be decanted from plant material and used to proceed. Record the preparation of the sample on the appropriate Drug Chemistry Worksheet form.

6.2.3.3. Add an equal volume of concentrated hydrochloric acid and mix.

6.2.3.4. Observe any color changes.

6.2.3.5. Add three volumes of chloroform and mix.

6.2.3.6. Allow phases to separate and observe the color in the chloroform (bottom) layer.

6.2.3.7. Record results and any observations.

6.2.4. Limitations

6.2.4.1. Wet or fresh plant material, old plant material and residues may need preparation as described in 6.2.3.1.1. or 6.2.3.2.1.

6.3. Cobalt Thiocyanate

6.3.1. Reacts with secondary and tertiary amines as well as some alkaloids to produce a blue color.

6.3.2. Selected characteristic results: Cocaine – blue
PCP – blue

6.3.3. Preparation: Dissolve 2.0 g cobalt (II) thiocyanate in 100 ml of deionized water.

6.3.3.1. Storage: Glass

6.3.3.2. Expiration: Stock container: Three years.
Use container: Three months

6.3.3.3. Lot number: Eight digit format year/month/day/CoSCN2/initials of preparer. Example: 20120131CoSCN2XXX

6.3.3.4. Positive Quality Control Check (PQCC):

6.3.3.4.1. Reference Material: Cocaine hydrochloride.
6.3.3.4.2. Acceptable result: A blue color is observed, i.e., Positive.

6.3.4. Procedure

6.3.4.1. Add 1-2 drops of the reagent to a clean spot well or a new test tube and observe any reaction or color produced.

6.3.4.1.1. If a significant color develops, take steps to ensure that the spot well is clean or use a new spot well or use a new test tube.
6.3.4.1.2. If the significant color formation persists prepare a new lot of reagent.

6.3.4.2. Add a small amount of sample to the reagent.

6.3.4.3. Observe any reaction or color produced.

6.3.4.4. Record observations.

6.4. Ferric Chloride

6.4.1. Reacts with phenols, enols, and GHB to produce color.

6.4.2. Selected characteristic results: Acetaminophen – blue-green
GHB - red/brown

6.4.3. Preparation: Dissolve 1.5 grams of ferric chloride in 29.0 milliliters of deionized water to produce a 5% w/v solution.

6.4.3.1. Storage: Glass

6.4.3.2. Expiration: Stock container: Three years
Use container: Three months

6.4.3.3. Lot number: Eight digit format year/month/day/FeCl3/initials of preparer. Example: 20120131FeCl3XXX

6.4.3.4. Positive Quality Control Check (PQCC):

6.4.3.4.1. Reference material: Acetaminophen
6.4.3.4.2. Acceptable result: A blue-green color is observed.

6.4.4. Procedure

6.4.4.1. Add 1-2 drops of the reagent to a clean spot well or a new test tube and observe any reaction or color produced.

6.4.4.1.1. If a significant color develops, take steps to ensure that the spot well is clean or use a new spot well or use a new test tube.
6.4.4.1.2. If the significant color formation persists prepare a new lot of reagent.

6.4.4.2. Add a small amount of sample to the reagent.

6.4.4.3. Observe any reaction or color produced.

6.4.4.4. Record observations.

6.5. Dille-Koppanyi (modified)

6.5.1. This color test reacts with barbiturates to produce a red-violet color.

6.5.2. Selected characteristic results: Barbiturates – red-violet.

6.5.3. Preparation

6.5.3.1. Dile-Koppanyi Paper

6.5.3.1.1. Dissolve 0.1 gram cobalt (II) acetate in 100 milliliters of methanol.

6.5.3.1.2. Add 0.2 milliliter glacial acetic acid.

6.5.3.1.3. Soak filter paper in the solution and allow to dry completely.

6.5.3.1.4. Cut filter paper into small pieces for use. (Approximate one inch squares suggested.)

6.5.3.1.5. Store filter paper in a wide mouth bottle with top.

6.5.3.1.6. Storage: Amber glass

6.5.3.1.7. Expiration: Use container: Three years

6.5.3.1.8. Lot number: Eight digit format year/month/day/DKPap/initials of preparer.
Example: 20120131DKPapXXX

6.5.3.1.9. Positive Quality Control Check (PQCC)

6.5.3.1.9.1. Reference material: Phenobarbital
6.5.3.1.9.2. Acceptable result: Red-violet color produced

6.5.3.2. 5% Isopropylamine

6.5.3.2.1. Mix 5 milliliters isopropylamine and 95 milliliters methanol.

6.5.3.2.2. Storage: Amber glass.

6.5.3.2.3. Expiration: Stock container: Three years
Use container: Three months

6.5.3.2.4. Lot number: Eight digit format year/month/day//initials of preparer.
Example: 20101231IPAm5%XXX

6.5.3.2.5. Positive Quality Control Check (PQCC)

6.5.3.2.5.1. Reference material: Phenobarbital
6.5.3.2.5.2. Acceptable result: Red-violet color produced

6.5.4. Procedure

6.5.4.1. Place a small amount of sample on a piece of the Dile-Koppanyi paper.

6.5.4.2. Press the sample onto the paper with a spatula (optional).

6.5.4.3. Place a drop of the 5 % Isopropylamine solution on the edge of the Koppanyi paper and tilt to allow the drop to meet the sample.

6.5.4.4. Record observations.

6.6. *para*-Dimethylaminobenzaldehyde (*p*DMAB)

6.6.1. This color test uses a filter paper soaked with the reagent. This test reacts with indoles (e.g., LSD), primary aromatic amines (e.g., procaine), and carbamates to produce colored intermediates.

6.6.2. Selected Characteristic Results: Carbamate – yellow
LSD – purple
Psilocin – dark purple
Procaine, Benzocaine – orange/yellow

6.6.3. Preparation: *p*DMAB Paper

6.6.3.1. Dissolve 1.0 gram of *p*DMAB in 100 milliliters of methanol.

6.6.3.2. Soak the filter paper in the solution and allow it to dry completely.

6.6.3.2.1. Cut filter paper into small pieces for use.

6.6.3.2.2. Storage: Amber glass

6.6.3.2.3. Expiration: Use container: Three years

6.6.3.2.4. Lot number: Eight digit format year/month/day/*p*DMAB/initials of preparer.
Example: 20120131*p*DMABXXX

6.6.3.3. Positive Quality Control Check (PQCC) QC check:

6.6.3.3.1. Reference material: LSD
6.6.3.3.2. Acceptable results: Purple color produced

6.6.4. Procedure

6.6.4.1. Place a small amount of sample on a piece of the *p*DMAB paper.

6.6.4.2. Press the sample onto the paper with a spatula. (optional)

6.6.4.3. Place a drop of methanol on top of the sample to help it dissolve into the paper.

6.6.4.4. Add a drop of concentrated hydrochloric acid to the filter paper by one of the following methods:

6.6.4.4.1. Adding the drop directly on the methanol spot.
6.6.4.4.2. Adding the acid drop to the edge of the paper and allowing the acid and methanol spots to meet (e.g., LSD and Psilocin.)
6.6.4.4.3. Allowing the fumes of the acid to contact the paper (e.g., procaine and benzocaine)

6.6.4.5. Heated air may be applied.

6.6.4.6. Record observations.

6.7. Froehde

6.7.1. This color test reacts with a wide range of aromatic compounds to produce colored intermediates.

6.7.2. Selected Characteristic Results: Heroin – purple
Morphine – purple
Bufotenine – yellow/brown
Oxycodone - yellow

6.7.3. Preparation: Prepare a 1% (w/v) solution of molybdic acid (or sodium molybdate) in concentrated sulfuric acid with heating and stirring.

6.7.3.1. Storage: Amber glass

6.7.3.2. Expriation: Stock container: One month
Use container: One month

6.7.3.3. Lot Number: Eight digit format year/month/day/Fro/initials of preparer. Example: 20120131FroXXX

6.7.3.4. Positive Quality Control Check (PQCC):

6.7.3.4.1. Reference material: Oxycodone
6.7.3.4.2. Acceptable results: Oxycodone produces a yellow color.

6.7.4. Procedure

6.7.4.1. Add 1-2 drops of the reagent to a clean spot well or a new test tube and observe any reaction or color produced.

6.7.4.1.1. If a significant color develops, take steps to ensure that the spot well is clean or use a new spot well or use a new test tube.

6.7.4.1.2. If the significant color formation persists prepare a new lot of reagent.

6.7.4.2. Add a small amount of sample to the reagent.

6.7.4.3. Observe any reaction or color produced.

6.7.4.4. Record observations.

6.8. Mecke

6.8.1. This color test reacts with a wide range of aromatic compounds to produce colored intermediates.

6.8.2. Selected Characteristic Results:

Bufotenine – brown to black/purple
Psilocin - green
Heroin HCl – green/blue
Hydrocodone bitartrate – dark blue
Methadone – green/brown
Oxycodone HCl - green

6.8.3. Preparation: Prepare a 1% (w/v) solution of selenious acid in concentrated sulfuric acid with stirring.

6.8.3.1. Storage:

6.8.3.2. Expiration: The expiration date for this reagent shall be one month after preparation.

6.8.3.3. Lot number: Eight digit format year/month/day/Mec/initials of preparer. Example: 20120131MecXXX

6.8.3.4. Positive Quality Control Check (PQCC):

6.8.3.4.1. Reference material: Oxycodone HCl

6.8.3.4.2. Acceptable results: Green color is produced

6.8.4. Procedure

6.8.4.1. Add 1-2 drops of the reagent to a clean spot well or a new test tube and observe any reaction or color produced.

6.8.4.1.1. If a significant color develops, take steps to ensure that the spot well is clean or use a new spot well or use a new test tube.

6.8.4.1.2. If the significant color formation persists prepare a new lot of reagent.

6.8.4.2. Add a small amount of sample to the reagent.

6.8.4.3. Observe any reaction or color produced.

6.8.4.4. Record observations.

6.9. Cobalt Nitrate

6.9.1. This color test reacts with GHB to produce a pink to violet color.

6.9.2. Selected Characteristic Results: GHB - pink to violet

6.9.3. Preparation: Dissolve 0.2 gram of cobalt (II) nitrate in 20 milliliters of water (1 % w/v solution.)

6.9.3.1. Storage:

6.9.3.2. Expiration: Stock container: Three years.
Use container: One year.

6.9.3.3. Lot number: Eight digit format year/month/day/CoNO3/initials of preparer. Example: 20120131CoNO3XXX

6.9.3.4. Positive Quality Control Check (PQCC):

6.9.3.4.1. Reference material: GHB
6.9.3.4.2. Acceptable results: pink to violet color

6.9.4. Procedure

6.9.4.1. Add a few drops of the reagent to a new test tube and observe any reaction or color produced.

6.9.4.1.1. If a significant color develops, use a new test tube.

6.9.4.1.2. If the significant color formation persists prepare a new lot of reagent.

6.9.4.2. Add 0.5 mL of the liquid sample.

6.9.4.3. Observe any reaction or color produced.

6.9.4.4. Record observations on the appropriate Drug Chemistry Worksheet form.

6.10. Zwikker

6.10.1. This color test reacts with barbiturates to produce a purple color that transfers to the organic layer of the reagent.

6.10.2. Selected Characteristic Results: Barbiturates – purple or bright green color that transfers to the organic layer

6.10.3. Preparation:

6.10.3.1. 0.5% Cupric Sulfate

6.10.3.1.1. Dissolve 0.12 gram cupric sulfate pentahydrate in 25 milliliters of deionized water.

6.10.3.1.2. Storage: Glass

6.10.3.1.3. Expiration: Stock container: Three years
Use container: One year

6.10.3.1.4. Lot number: Eight digit format year/month/day/CuSO4/initials of preparer. Example: 20120131CuSO4XXX

6.10.3.1.5. Positive Quality Control Check (PQCC):

6.10.3.1.5.1. Reference material: Phenobarbital
6.10.3.1.5.2. Acceptable results: Phenobarbital produces a purple color that transfers to the organic layer.

6.10.3.2. 5% Pyridine

6.10.3.2.1. Add 1 milliliter of pyridine to 19 milliliters of chloroform.

6.10.3.2.2. Storage: Amber glass

6.10.3.2.3. Expiration: Stock Container: Three years
Use container: One year

6.10.3.2.4. Lot number: Eight digit format year/month/day/Pyr5%/initials of preparer. Example: 20120131Pyr5%XXX

6.10.3.2.5. Positive Quality Control Check (PQCC):

6.10.3.2.5.1. Reference material: Phenobarbital

6.10.3.2.5.2. Acceptable results: Phenobarbital produces a purple color that transfers to the organic layer.

6.10.3.3. Procedure

6.10.3.3.1. Place a small amount of sample in a culture tube.

6.10.3.3.2. Add a drop of 0.5 % cupric sulfate and observe any reaction or color change.

6.10.3.3.3. Add a drop of 5 % pyridine and observe any reaction or color change.

6.10.3.3.4. Record observations.

6.11. Simon's Test (Modified Sodium Nitroprusside)

6.11.1. This color test reacts with secondary amines to produce a blue-violet color and it reacts with primary amines to produce a slow pink to cherry red color.

6.11.2. Selected characteristic results: Methamphetamine and secondary amines: blue-violet
Amphetamine and primary amines : slow pink to cherry red

6.11.3. Preparation:

6.11.3.1. 1 % (w/v) Sodium Nitroprusside / 10 % by volume of acetaldehyde

6.11.3.1.1. Dissolve 0.9 gram of sodium nitroprusside in 90 milliliters of water.

6.11.3.1.2. Add 10 milliliters of acetaldehyde.

6.11.3.1.3. Storage: Refrigerate in amber glass

6.11.3.1.4. Expiration: Stock container: one month
Use container: One month

6.11.3.1.5. Lot number: Eight digit format year/month/day/SNP/initials of preparer. Example: 20120131SNPXXX

6.11.3.1.6. Positive Quality Control Check (PQCC)

6.11.3.1.6.1. Reference material: Methamphetamine
6.11.3.1.6.2. Acceptable results: Blue-violet color

6.11.3.2. 2 % (w/v) Sodium Carbonate

6.11.3.2.1. Dissolve 2 grams of sodium carbonate in 100 milliliters of water.

6.11.3.2.2. Storage: Closed container

6.11.3.2.3. Expiration: Stock container: Three years
Use container: One year

6.11.3.2.4. Lot number: Eight digit format year/month/day/Na2CO3/initials of preparer. Example: 20120131Na2CO3XXX

6.11.3.2.5. Positive Quality Control Check (PQCC)

6.11.3.2.5.1. Reference material: Methamphetamine
6.11.3.2.5.2. Acceptable results: Blue-violet color

6.11.3.3. Procedure

6.11.3.3.1. Place a small amount of sample in a culture tube or clean spot well and add one drop of the sodium nitroprusside reagent then add 2 drops of 2 % sodium carbonate.

6.11.3.3.2. Observe any reaction or color produced.

6.11.3.3.3. Record observations.

7. Limitations - See above and the Drug Chemistry Unit Technical Procedure for Drug Chemistry Analysis.

8. Safety – Refer to CCBI Crime Laboratory Safety Manual.

9. References

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9.1.3. Clarke, E.G.C. and R.G. Todd, eds. *Isolation and Identification of Drugs*. 1st Edition. London: Pharmaceutical Press, 1969, 129-130, 1183.

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9.1.6. Feigel, Fritz. *Spot Tests in Organic Analysis*. Elsevier Publishing Co.: 1956, 251.

9.1.7. Johns, S.H. "Spot Tests: A Color Chart Reference for Forensic Chemists." *Journal of Forensic Sciences*, Volume 24. Issue 3 (July, 1979): 631-649.

9.1.8. Jungreis, Ervin. *Spot Test Analysis - Clinical, Environmental, Forensic, and Geochemical Applications*, New York: John Wiley & Sons, 1985, 80.

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9.1.10. Liu, Ray H. and Daniel E. Gadzala. *Handbook of Drug Analysis: Applications in Forensic and Clinical Laboratories*. Washington, D.C.: American Chemical Society, 1997: 58.

9.1.11. Toole, K.E. et. al. "Color Tests for the Preliminary Identification of Methcathinone and Analogues of Methcathinone." *Microgram Journal*. Volume 9, Number 1: 27-32.

9.1.12. O'Neal, C.L. et. al. "Validation of Twelve Chemical Spot Tests for the Detection of Drugs of Abuse." *Forensic Science International.* Volume 109 (2000): 189-201.

10. Records

10.1. Prepared reagent log
10.2. Drug Chemistry worksheets

Revision History		
Effective Date	Version Number	Reason
1/1/2013	1	ISO Compliance
2/16/15	2	Added glacial acetic acid, ACS grade to commercial reagents

8: Ultraviolet Spectroscopy

1. **Purpose / Scope** - This procedure provides direction for the initial setup, performance checks and usage of the ultraviolet spectrometer in the Drug Chemistry Unit of the Raleigh/Wake City-County Bureau of Identification.

2. **Definitions**

   2.1. Performance verification – The initial confirmation of the reliability of a previously or externally validated method or instrument.
   2.2. Quality control check – Periodic confirmation of the reliability of equipment, instrumentation, and/or reagents.
   2.3. Reference material – Material sufficiently homogenous and stable with reference to specified properties, which has been established to be fit for its intended use in measurement or in examination of nominal properties.
   2.4. Reference standard - Measurement standard designated for the calibration of other measurement standards (reference standards or equipment.)

3. **Abbreviations**

   3.1. Refer to the Drug Chemistry Unit Technical Procedure for Drug Chemistry Analysis
   3.2. QCC – Quality Control Check
   3.3. UV – Ultraviolet Spectrometer

4. **Equipment, Materials and Reagents**

   4.1. Equipment

   4.1.1. Perkin-Elmer Lambda 20 Ultraviolet Spectrometer, fixed 2 nm spectral bandwidth with printer and UV WinLab Software.

   4.2. Reference Standard

   4.2.1. Holmium oxide glass filter

   4.3. Materials

   4.3.1. Gloves
   4.3.2. Eye protection
   4.3.3. Laboratory coat
   4.3.4. Graduated cylinder
   4.3.5. Quartz UV cells, 10 mm lightpath

4.3.6. Pipettes with bulb
4.3.7. Spatula
4.3.8. Water, deionized

4.4. Commercial Reagents

4.4.1. Hydrochloric acid, ACS grade
4.4.2. Sodium hydroxide, ACS grade
4.4.3. Organic solvents, ACS and spectrophotometric grade

5. Prepared Reagents – Refer to the Drug Chemistry Unit Technical Procedure for Receipt and Quality Assurance of Laboratory Supplies, Reagents, Reference Collections, Reference Standards and Reference Materials for additional instructions regarding prepared reagents.

5.1.1. Hydrochloric Acid, 0.05N

5.1.1.1. Add 1ml of concentrated (12N) hydrochloric acid to 250 ml water and mix.

5.1.1.2. Storage:
Stock container: closed container in acid storage location
Use container: closed container or wash bottle on benchtop or in fume hood

5.1.1.3. Expiration:
Stock container: Three years
Use container: Six months

5.1.1.4. Lot number: Eight digit year format year/month/day/HCl0.05N/initials of preparer. Example: 20120131HCl0.05NXXX

5.1.1.5. Quality control check: acidic to litmus paper.

6. Standards and Controls

6.1. A UV logbook shall be maintained near the instrument. The UV logbook shall contain the UV Activity Log, the UV Daily QCC Log, the UV Maintenance Log and any manufacturer's certificates, performance verification documentation, QCC printouts and maintenance documentation.

6.2. When the UV has been placed out of service (e.g., maintenance, malfunction, leaving direct control of the Laboratory), a Daily QCC must be successfully performed prior to placing the instrument back in service, refer to 6.5.

6.3. The Drug Chemist shall record any malfunctions or error messages in the UV Activity Log, notify the Drug Chemistry Technical Leader of any malfunctions or error messages and place the instrument out of service by marking the UV Activity Log "Out of Service."

6.4. The Drug Chemistry Technical Leader shall examine the effect(s), if any, of a malfunction or error message on analysis results and implement the CCBI Laboratory Procedure for Corrective and Preventive Action as required.

6.5. Daily Quality Control Check

6.5.1. The daily QCC shall be performed daily, prior to casework, and following any shutdown by a Drug Chemist using a holmium oxide glass filter reference standard each day the instrument is in use.

6.5.2. With the cell holders empty, close the sample compartment cover completely.

6.5.3. ~~From the standby display "INPUT > <", select method 505 by pressing [Method] followed by [5], [0], [5] and [Enter].~~
Open the Perkin Elmer UV Winlab software on the computer. Select (double click) the holmium oxide method. This will open a UV Winlab Run window. Method parameters are as listed:

Method: Holmium Oxide ~~505~~
Data Collection:
Ordinate mode in absorbance
Scan range: 210 – 700 nm
Data Interval ~~Smooth~~: ~~0~~ 1 nm
~~Autozero~~
Cycle time: 0.1 minute
~~Ordinate max: 3.00 abs~~
~~Scale: 50.0 nm/cm~~
Speed: 30 nm/min
Lamp: UV and VIS
Cycle: 1
Processing:
Threshold: 0.1 abs
Display: All peaks
Interpolation: On

~~6.5.4.~~ Designate the type of sample in the "type" column and define your NQCC filename and DQCC filename in the "sample ID" boxes. Select "start". This will prompt you to remove samples and press OK to perform a 100% T / 0A correction (Autozero).
Click OK. ~~At the "Autozero" prompt, press [Start} to begin the autozero process.~~

~~6.5.5.~~ Once the autozero is complete the software will prompt you to insert and collect a blank. Select OK.
~~At the "Sample 1" prompt, insert the holmium oxide reference material cell into the sample cell holder (front) and press [Start].~~

~~6.5.6.~~ When the blank is finished the software will prompt you to insert the holmium oxide standard in the instrument and to press OK to begin data collection. Select OK. Select output and use the report template "Holm.3" Select "Preview" to preview the report and select the printer icon to print the holmium oxide NQCC and DQCC.
~~At the next prompt press [Stop] and wait for the printer to complete the print job.~~

6.5.7. Compare the peaks identified to the following table. Peaks shall be identified near the wavelengths listed in column A within the range specified in column B. Record the results in the UV Daily QCC Log.

Perkin-Elmer Lambda 20, fixed 2.0 nm SBW Spectral Bandwidth = 2 nm Acceptable Range is ± 1.0 nm	
Peak(nm)	**Acceptable Range (nm)**
279.3	**278.3 – 280.3**
360.8	**359.8 – 361.8**
459.9	**458.9 – 460.9**
536.4	**535.4 – 537.4**

6.5.8. Record the Daily QCC on the UV Daily QCC log with initials, date, observed peak, difference from expected peak, results (pass/fail), and any comments.

6.5.9. If any peaks are outside of the acceptable range, the instrument shall be placed out of service by marking the UV activity log "Out of Service." Notify the Drug Chemistry Technical Leader.

6.5.9.1. The Drug Chemistry Technical Leader shall correct any problems with the instrument or request service. The Drug Chemistry Technical Leader shall examine the effect(s), if any, on analysis results and implement the CCBI Laboratory Procedure for Corrective and Preventive Action as required.

6.5.9.2. The Daily Quality Control check must be successfully completed prior to placing the instrument back in service.

6.5.10. Label the printouts with initials, date and holmium oxide glass filter identifier and record the printouts in the UV Logbook.

6.6. Negative Quality Control Checks are performed with each sample analyzed~~, refer to 8.4.6~~.

6.7. Performance Verification for New Instruments

6.7.1. New instruments shall be installed by a manufacturer representative or approved vendor according to the manufacturer's specifications.

6.7.2. Prior to use, a successful Daily QCC shall be performed, refer to 6.5.

6.7.3. Prior to use, collect UV spectra for three controlled substance reference materials.

6.7.3.1. Compare the absorbance peaks and spectra of each to previously obtained spectra for the reference materials or published data. The data obtained must be substantially the same as the previously obtained data or published data for each compound.

6.7.4. Label the instrument printouts with the lot numbers of the reference materials, initials and date. Record the Performance Verification in the UV Logbook and place the printouts in the UV Logbook.

6.7.5. The performance verification must be reviewed and approved by the Drug Chemistry Technical Leader prior to the instrument being used for casework. The Drug Chemistry Technical Leader shall record the review and approval in the UV logbook.

7. Maintenance

7.1. Record all maintenance, other than cleaning, in the UV Maintenance Log at the time it is performed with the name of the person performing the maintenance or repairs, the initials of the Drug Chemist recording the maintenance or repairs, the date, a description of the maintenance or repairs and a list of any parts replaced.

7.2. Place the instrument out of service prior to performing any maintenance other than daily cleaning by marking the UV activity log "Out of Service." When the instrument is ready to be returned to service, mark the UV activity log "Back in Service."

7.3. Annual preventive maintenance will be performed by an approved vendor.

7.4. Required post-maintenance checks prior to placing the instrument back in service: Daily QCC, refer to 6.5.

8. Procedure

8.1. Instrument Startup

8.1.1. Open the sample compartment cover and ensure that the beam paths are free.

8.1.2. Close the sample compartment cover and switch on the instrument at the power switch. Ensure that ~~Switch on~~ the printer is on.

8.1.3. Wait for the standby display to appear: INPUT > <

8.1.4. Allow the lamps to warm up and stabilize for a minimum of ten minutes.

8.1.5. A successful daily QCC must be performed prior to casework following any shutdown, refer to 6.5.

8.2. Instrument Shutdown – the instrument should be off when not in use.

~~8.2.1.~~ If the instrument is not at standby, return to standby by pressing [Stop] or [Param].

8.2.2. Open the sample compartment cover and ensure that all samples and cells are removed from the sample compartment.

8.2.3. Clean any spilled material with a lintless paper wipe. If necessary, use a soft cloth and mild detergent solution to clean followed by wiping with a clean cloth wet with water and then drying with lintless paper wipes.

8.2.4. Close the sample compartment cover.

8.2.5. Switch the instrument off and switch the printer off.

8.3. UV Cell Handling

8.3.1. Handle only the frosted sides.

8.3.2. Prior to use, inspect the UV cells to ensure that they are clean and dry without scratches or fingerprints. If necessary, rinse the cells with 0.05 N HCl, deionized water or other suitable solvent. Wipe dry with a lintless paper wipe.

8.3.3. Fill cells to within one quarter inch of the top and ensure that no bubbles cling to the inner surface of the cell.

8.3.4. Upon completion of analysis, rinse cells thoroughly with the analysis solvent followed by thorough rinsing with deionized water.

8.3.4.1. Dry the cells with lintless paper wipes.

8.3.4.2. Store the cells in a closed, padded storage box.

8.4. Sample Qualitative Measurement

8.4.1. Fill two matching UV cells with 0.05 N HCl or other suitable solvent, refer to 8.3. Solvents other than 0.05 N HCl may be used due to pH shifts of absorbance maxima and peak absorbance in non-aqueous media. Most literature references use 0.05 N HCl.

8.4.2. Open the Perkin Elmer UV Winlab software on the computer. From the UV Winlab explorer window, select (double click) "sample" as the method. Method parameters are as listed:

Method: Sample
Data Collection:
Ordinate mode in absorbance
Scan range: 210 – 350 nm
Data Interval: 1 nm
Cycle time: As fast as possible
Speed: 30 nm/min
Lamp: UV and VIS
Cycle: 1
Processing:
Threshold: 0.01 abs
Display: All peaks
Interpolation: On

~~Place one UV cell in the reference cell holder (rear) and one UV cell in the sample cell holder (front) with the non-frosted sides of the cells in the light path.~~

~~8.4.3.~~ Select "Sample Info." Designate the type of sample in the "type" column and define your NQCC filename and sample filename in the "sample ID" boxes. Insert rows as needed. ~~Close the sample compartment cover completely.~~

8.4.4. Select "start". This will prompt you to remove samples and press OK to perform a 100% T / 0A correction (Autozero). Click OK.
~~From the standby display "INPUT > <", select method 502 by pressing [Method] followed by [5], [0], [2] and [Enter]. Methods 501, 503 or 504 may also be used, primarily to adjust the printed scale.~~ ~~Method parameters are as listed:~~

~~**8.4.4.1.** Method 501~~
~~Ordinate mode in absorbance~~
~~Scan range: 210 – 350 nm~~
~~Smooth: 0 nm~~
~~Autozero~~
~~Cycle time: 0.1 minute~~
~~Ordinate max: 3.00 abs~~
~~Scale: 10.0 nm/cm~~
~~Speed: 30 nm/min~~
~~Lamp: UV and VIS~~
~~Cycle: 1~~

~~Threshold: 0.1 abs~~

~~**8.4.4.2.** Method 502~~

~~Ordinate mode in absorbance~~
~~Scan range: 210 – 350 nm~~
~~Smooth: 2 0 nm~~
~~Autozero~~
~~Cycle time: 0.1 minute~~
~~Ordinate max: 2.00 abs~~
~~Scale: 20.0 10.0 nm/cm~~
~~Speed: 240 30 nm/min~~
~~Lamp: UV and VIS~~
~~Cycle: 1~~
~~Threshold: 0.1 abs~~

~~**8.4.4.3.** Method 503~~

~~Ordinate mode in absorbance~~
~~Scan range: 210 – 350 nm~~
~~Smooth: 2 0 nm~~
~~Autozero~~
~~Cycle time: 0.1 minute~~
~~Ordinate max: 1.00 abs~~
~~Scale: 20.0 10.0 nm/cm~~
~~Speed: 240 30 nm/min~~
~~Lamp: UV and VIS~~
~~Cycle: 1~~
~~Threshold: 0.1 abs~~

~~**8.4.4.4.** Method 504~~

~~Ordinate mode in absorbance~~
~~Scan range: 210 – 350 nm~~
~~Smooth: 2 0 nm~~
~~Autozero~~
~~Cycle time: 0.1 minute~~
~~Ordinate max: 0.50 abs~~
~~Scale: 20.0 10.0 nm/cm~~
~~Speed: 240 30 nm/min~~
~~Lamp: UV and VIS~~
~~Cycle: 1~~
~~Threshold: 0.1 abs~~

8.4.5. Once the autozero is complete the software will prompt you to insert and collect a blank. Place one UV cell containing solvent in the reference cell holder (rear) and one UV cell containing solvent in the sample cell holder (front) with the non-frosted sides of the cells in the light path. Close the sample compartment cover completely. Select OK.

~~Press [Start] and at the "Autozero" prompt, press [Start}] to begin the autozero process.~~

~~**8.4.6.**~~When the NQCC is finished the software will prompt you to insert the sample in the instrument. Add the sample to the cell and mix, refer to 8.3. Select OK to begin data collection. When the data collection is complete, select output and use the report template "Holm3." Select "preview" to view the reports and select the printer icon to print the NQCC and sample.
~~Press [Start] and at the "Sample 1" prompt press [Start] to perform a blank sample scan as a negative QCC~~

8.4.7. The negative QCC must be free from any significant absorbance.

8.4.7.1. If a significant absorption is observed, clean the cell, refer to 8.3, and repeat the measurement. Additional steps to correct the problem may include preparing or obtaining a new solvent container. Print, label and store the printout(s) in the UV logbook.

8.4.7.2. If the problem cannot be corrected, place the instrument out of service by marking the activity log "Out of Service" and notify the Drug Chemistry Technical Leader.

8.4.7.3. The Drug Chemistry Technical Leader shall correct any problems with the instrument or request service. The Drug Chemistry Technical Leader shall examine the effect(s), if any, on analysis results and implement the CCBI Laboratory Procedure for Corrective and Preventive Action as required.

8.4.7.4. The daily QCC, refer to 6.5., must be successfully completed prior to placing the instrument back in service.

8.4.8. Record the negative QCC solvent and results (pass / fail) on the UV Activity Log.

~~**8.4.9.** At the next "Sample 2" prompt, remove the cell from the sample cell holder (front), add the sample to the cell and mix, refer to 8.3.~~

~~**8.4.10.** Place the sample cell in the sample cell holder and press [Start].~~

~~**8.4.11.** At the next prompt press [Stop] and wait for the printer to complete the print job.~~

8.4.12. Label the printouts with initials, date, case number, item number and any other information necessary to uniquely identify the sample and place the printouts in the case record.

8.4.13. Record the method, solvent, solvent lot number if a prepared reagent and results on the appropriate Drug Chemistry Worksheet form.

8.4.14. Record the date, initials, method, sample identification, and any comments on the UV Activity Log.

8.4.15. Absorbance peaks and the spectrum may be compared to reference material or published values. Record the appropriate identifying information in the case record, e.g., lot number, literature source.

9. Safety – Refer to CCBI Crime Laboratory Safety Manual

10. References

10.1. Skoog, D. A., Holler, F. J., Nieman, T. A., *Principles of Instrumental Analysis*. 5th ed. Harcourt, Brace & Company, 1998: 299-354.

10.2. Anthony C. Moffat et al., *Clarke's Analysis of Drugs and Poisons*. 4th ed. London, UK: Pharmaceutical press, 2011.

10.3. *Lambda 20 UV/Vis Installation, Maintenance, System Description Part Number 0993-5055*, Revision A. USA: Perkin-Elmer Corporation, 1996.

10.4. *Lambda 20 UV/Vis Operation and Parameter Description Part Number 0993-5056*, Revision A. USA: Perkin-Elmer Corporation, 1996.

10.5. *Standard Practice for Monitoring the Calibration of Ulraviolet – Visible Spectrophotometers whose Spectral Bandwidth does not Exceed 2nm*, ASTM:E925-09.

10.6. Allen, D. W., *Holmium Oxide Glass Wavelength Standards*. J. Res. Natl. Inst. Stand. Technol. 112, 303-306 (2007).

11. Records

11.1. UV Activity Log
11.2. UV Daily QCC Log
11.3. UV Maintenance Log

Revision History		
Effective Date	Version Number	Reason
1/1/13	1	ISO Compliance
11/26/13	2	Updated UV Procedures and Daily QCC Log
3/31/16	3	Updated for use of WinLab UV software.

9: Technical Procedure for Infrared Spectroscopy

1. **Purpose / Scope** - This procedure provides direction for the initial setup, performance checks and usage of infrared spectrometers Spectrometer in the Drug Chemistry Unit of the Raleigh/Wake City-County Bureau of Identification.

2. **Definitions**

   2.1. Performance verification - The initial confirmation of the reliability of a previously or externally validated method or instrument.
   2.2. Quality control check - Periodic confirmation of the reliability of equipment, instrumentation, and/or reagents.
   2.3. Reference Material – Material sufficiently homogeneous and stable with reference to specified properties, which has been established to be fit for its intended use in measurement or in examination of nominal properties.

3. **Abbreviations**

   3.1. Refer to the Drug Chemistry Unit Technical Procedure for Drug Chemistry Analysis
   3.2. FTIR or IR – Fourier Transform Infrared Spectrometer
   3.3. ATR – Attenuated Total Reflectance
   3.4. P – Pink
   3.5. B - Blue

4. **Equipment, Materials, and Reagents**

   4.1. Equipment

      4.1.1. Frontier Fourier Transform Infrared Spectrometer with Universal Attenuated Total Reflectance Sampling Accessory and Spectrum Software version 7 with printer

   4.2. Reference Material

      4.2.1. Frontier FTIR Filter Wheel Polystyrene

   4.3. Materials

      4.3.1. Spatula
      4.3.2. Water, deionized
      4.3.3. Desiccant packs, PerkinElmer part numbers N0171159 or L1250311
      4.3.4. Fuses - 2A, 250V, PerkinElmer part number 04970839

   4.4. Commercial Reagents

4.4.1. Methanol or other suitable organic solvent, Optima or ACS grade

5. Standards and Controls

5.1. An IR logbook shall be maintained near the instrument. The IR logbook shall contain the IR Activity Log, IR Daily QCC Log, the IR Maintenance Log and any manufacturer's certificates, performance verification documentation, QCC printouts and maintenance documentation.

5.2. When the IR has been placed out of service (e.g., maintenance, malfunction, leaving direct control of the Laboratory), a Daily Quality Control Check must be successfully performed prior to placing the instrument back in service, refer to 5.6.

5.3. The Drug Chemist shall record any malfunctions or error messages in the IR Activity Log, notify the Drug Chemistry Technical Leader of any malfunctions or error messages and place the instrument out of service by marking the IR Activity Log "Out of Service."

5.4. The Drug Chemistry Technical Leader shall examine the effect(s), if any, of a malfunction or error message on analysis results and implement the CCBI Laboratory Procedure for Corrective and Preventive Action as required.

5.5. Negative Quality Control Check

5.5.1. Prior to collecting each sample scan, collect a negative QCC scan, using the procedure in Section 7, with no sample present.

5.5.2. Evaluate the acquired spectrum, it must be free from any significant peaks.

5.5.2.1. If significant peaks are present, repeat the cleaning of the crystal, background collection and the negative QCC scan with no sample present.

5.5.2.2. If the presence of significant peaks persists, mark the instrument "Out of Service" on the IR activity log. Notify the Drug Chemistry Technical Leader.

5.5.2.2.1. Print all negative QCC's with significant peaks, mark with initials, date and instrument serial number. Place the printouts in the IR logbook.

5.5.2.2.2. The Drug Chemistry Technical Leader shall examine the effect(s), if any, on analysis results and implement the CCBI Laboratory Procedure for Corrective and Preventive Action as required.

5.5.2.2.3. The Drug Chemistry Technical Leader shall either correct the problem or schedule service.

5.5.2.2.4. A successful Daily Quality Control Check, refer to 5.6., shall be performed prior to placing the instrument back in service.

5.5.3. Record all negative QCC's and results (pass/fail) on the IR activity log.

5.5.4. Print the negative QCC spectrum and mark it with initials, date, instrument serial number, case number, item number and any other information needed to uniquely identify the preceded sample and place in the case record.

5.6. Daily Quality Control Checks

5.6.1. The desiccant check and the daily polystyrene quality control check shall be performed daily, prior to casework and following any shutdown, by a Drug Chemist each day the instrument is in use.

5.6.2. Desiccant check - observe the desiccant indicator on top of the instrument.

5.6.2.1. Observe the desiccant indicator on top of the instrument. The sector marked "10" should be blue in color. If the "10" sector is pink the desiccant should be replaced soon, refer to 7.2.2 biannual maintenance.

5.6.2.2. If any other sector is pink, place the instrument out of service by marking the IR Activity Log "Out of Service." The desiccant must be replaced, refer to refer to 6.3.2 biannual maintenance.

5.6.2.3. Record the Desiccant check in the IR Daily QCC Log by recording the color of sector "10" as pink (P) or blue (B).

5.6.3. Daily Polystyrene Quality Control Check - collect a polystyrene scan, using the procedure in Section 7., with the filter wheel polystyrene in the beam path.

5.6.3.1. After completing the negative QCC, place the polystyrene in the beam path:

5.6.3.1.1. Choose "Scan and Instrument Setup."
5.6.3.1.2. Choose "Beam" tab.
5.6.3.1.3. Double click on the picture of the filter wheel.
5.6.3.1.4. Choose "Polystyrene."
5.6.3.1.5. Choose "OK."
5.6.3.1.6. Choose "Apply."

5.6.4. Choose "Label Peaks" hot key to display peak data.

5.6.5. Print the acquired spectrum and mark it with initials, date, instrument serial number, and "Daily QCC – internal polystyrene."

5.6.6. Remove the polystyrene from the beam path:

5.6.6.1.1. Choose "Scan and Instrument Setup" hotkey.
5.6.6.1.2. Choose "Beam" tab.
5.6.6.1.3. Double click on the picture of the filter wheel.
5.6.6.1.4. Choose "None."
5.6.6.1.5. Choose "OK."
5.6.6.1.6. Choose "Apply."

5.6.7. Compare the peaks identified to the list below. Peaks must be identified near the wavenumbers listed within +/- 1.0 cm^{-1}:

5.6.7.1. 3082.22 cm^{-1}
5.6.7.2. 3060.14 cm^{-1}
5.6.7.3. 1601.38 cm^{-1}
5.6.7.4. 1583.04 cm^{-1}
5.6.7.5. 1028.42 cm^{-1}

5.6.8. Record the observed peak on the IR Daily QCC Log.

5.6.8.1. If any peaks are outside of the acceptable range, place the instrument out of service by marking the IR Activity Log "Out of Service."

5.6.8.1.1. The Drug Chemistry Technical Leader shall correct any problems with the instrument or request service. The Drug Chemistry Technical Leader shall examine the effect(s), if any, on analysis results and implement the CCBI Laboratory Procedure for Corrective and Preventive Action as required.

5.6.8.1.2. The daily polystyrene QCC must be successfully completed prior to placing the instrument back in service.

5.6.9. Place all QCC spectra in the IR Logbook.

5.7. Performance Verification for New Instrument

5.7.1. New FT-IR instruments shall be installed by a manufacturer representative or approved vendor according to the manufacturer's guidelines.

5.7.1.1. The installation shall include a demonstration of traceability of the instrument using polystyrene traceable reference material according to the manufacturer's requirements and a demonstration of the traceability of the internal polystyrene using the universal ATR sampling accessory according to the requirements in 5.6.7.

5.7.2. Prior to use, collect spectra of three controlled substance reference materials, e.g., methamphetamine, phentermine, and cocaine base, according to the procedure, refer to

Section 7. Compare the spectra to previously obtained spectra for the reference materials or published data. The data obtained must be substantially the same as the previously obtained data or published data for each compound.

5.7.3. Label the instrument printouts with the lot numbers of the reference materials, initials and date. Record the Performance Verification in the IR Logbook and place the printouts in the IR Logbook.

5.7.4. The performance verification must be reviewed and approved by the Drug Chemistry Technical Leader prior to the instrument being used for casework. The Drug Chemistry Technical Leader shall record the review and approval in the IR logbook.

6. Maintenance

6.1. Record all maintenance, other than cleaning, in the IR Maintenance Log at the time it is performed with the name of the person performing the maintenance or repairs, the initials of the Drug Chemist recording the maintenance or repairs, the date, a description of the maintenance or repairs and a list of any parts replaced.

6.2. Place the instrument out of service prior to performing any maintenance other than daily cleaning by marking the IR activity log "Out of Service." When the instrument is ready to be returned to service, mark the IR activity log "Back in Service."

6.3. Suggested Routine Maintenance Schedule

6.3.1. Annual Maintenance

6.3.1.1. Yearly preventive maintenance and demonstration of NIST traceability of the instrument using polystyrene traceable reference material will be performed by an approved vendor.

6.3.1.2. A scan of a Traceable Reference Material polystyrene film shall be collected annually during the annual preventive maintenance. The Drug Chemistry Technical Leader shall compare the current Daily QCC spectrum to the Traceable Reference Material spectrum for the peaks listed in 5.6.7. The acceptable range is ± 0.5 cm^{-1} of the nominal value.

6.3.1.3. Maintain the spectra in the IR Logbook.

6.3.1.4. Required post-maintenance checks prior to placing the instrument back in service: Negative QCC, refer to 5.5
Daily QCC, refer to 5.6

6.3.2. Biannual Maintenance

6.3.2.1. Dessicant packs shall be changed at approximately six month intervals, or sooner when needed.

6.3.2.2. Remove the ATR accessory from the sample compartment. Pull lever underneath to release before removing.

6.3.2.3. Loosen the two captive screws securing the desiccant cover.

6.3.2.4. Open the cover and remove all the desiccant packs, noting how they were installed.

6.3.2.5. Place the six packs of desiccant in the desiccant holder. Ensure that when the packs have been installed they do not extend above the level of the black rubber purge seal.

6.3.2.5.1. The desiccant packs may be new or recharged packs. Recharge desiccant packs immediately before use by placing them in an oven at approximately 250 °C for approximately 8 hours and cooling in a desiccator.

6.3.2.6. Close the cover and tighten the screws.

6.3.2.7. Carefully refit the ATR accessory into the sample compartment area.

6.3.2.8. Change the desiccant alert clock in the software:

6.3.2.8.1. Choose “Scan and Instrument Setup” (hotkey).
6.3.2.8.2. Choose “Adjustment Toolbox” (“Hammer/wrench” icon).
6.3.2.8.3. Choose “Maintenance” (“Oil can” icon).
6.3.2.8.4. Choose “Changed” checkbox and the date will reset to the current day.
6.3.2.8.5. 180 days is the typical interval for the counter.

6.3.2.9. Required post-maintenance checks prior to placing the instrument back in service: Negative QCC, refer to 5.5
Daily QCC, refer to 5.6

6.3.3. Cleaning

6.3.3.1. If necessary, clean the outside of the instrument with a damp cloth. Mild detergent may be used.

6.3.3.2. Required post-maintenance checks prior to placing the instrument back in service: None.

6.3.3.3. Cleaning need not be recorded in the IR Maintenance Log.

6.4. Non-Routine Maintenance

6.4.1. Fuse change

6.4.1.1. If repeated fuse failures occur, there is an electrical fault. Disconnect the power supply and notify the Drug Chemistry Technical Leader for service scheduling. Place the instrument out of service by marking the IR activity log "Out of Service."

6.4.1.2. Switch off the power, wait for at least 60 seconds, and disconnect the power cable.

6.4.1.3. A spare fuse is stored in the left side of the fuse drawer located at the rear of the instrument between the power switch and the power socket.

6.4.1.4. Open the fuse drawer so that it swings down over the power socket.

6.4.1.5. Remove the old fuse located on the right side.

6.4.1.6. Remove the spare fuse located on the left side and fit it into the slot on the right.

6.4.1.7. Close the fuse drawer.

6.4.1.8. Connect the power cable and switch the instrument on.

6.4.2. Required post maintenance checks prior to placing the instrument back in service:
Negative QCC, refer to 5.5
Daily QCC, refer to 5.6.

7. Procedure

7.1. Instrument Settings

7.1.1. Scan range – 4000.00 to 550.00 cm^{-1}
7.1.2. Number scans – 4
7.1.3. Resolution – 4 cm^{-1}
7.1.4. Atmoshperic (CO_2/H_2O) Suppression - On
7.1.5. Diamond/Zinc Selenide crystal – one bounce

7.2. Shutdown/Startup

7.2.1. The power switch to the infrared instrument shall be left ON at all times to ensure the optics stay warm and excess moisture does not build up in the instrument.

7.2.2. The software and computer may be shut down at the end of each business day.

7.2.3. If a shutdown occurs, i.e., power failure, correct operation shall be demonstrated prior to casework by performing a Daily QCC, refer to 5.6.

7.3. Procedure for solid and liquid samples

7.3.1. Clean the ATR sampling accessory crystal by wiping with a lintless paper wipe wetted using water or an organic solvent while firmly holding the accessory in place. Wipe dry with a lintless paper wipe while firmly holding the accessory in place or allow to ~~and~~ air dry completely. When cleaning, do not apply liquids directly to the top of the ATR sampling accessory, volatiles may be introduced into the beam path and interfere with sample analysis.

7.3.2. Collect a background scan daily and additionally as prompted by the instrument.

7.3.3. Perform and evaluate the negative QCC as described in 5.6.

7.3.4. Place approximately 1 milligram or 1 drop of sample onto the ATR crystal.

7.3.5. Apply force using the ATR force arm to ensure good contact between the sample and the surface of the crystal, taking care to not apply excessive force. The instrument display indicates the force applied and sounds an alarm when excessive force is detected.

7.3.6. Scan to acquire data, a macro may be used to collect the spectrum and perform data handling.

7.3.6.1. The amount of sample on the ATR crystal may be adjusted to produce better a quality (too much sample indicated by intense, broad peaks; too little sample indicated by weak, noisy peaks) spectrum and the scan repeated.

7.3.6.2. Perform the following functions on the collected spectrum: auto baseline, normalize, ATR correction.

7.3.7. Print the acquired spectrum.

7.3.7.1. Label the spectrum with the initials, date, instrument serial number, case number, item number and any other information needed to uniquely identify the sample, e.g. extraction.

7.3.7.2. Record the acquired spectrum in the case record.

7.3.8. Spectral subtractions may be performed using the instrument software and spectra of primary or secondary reference material identified with supplier and lot number.

7.3.8.1. Print the resulting subtracted spectrum and any spectra used for subtraction.

7.3.8.2. Label the spectrum with the initials, date, instrument serial number, case number, item number, a marking to indicate that it was produced from a subtraction and any other information needed to uniquely identify the sample.

7.3.8.3. Label the spectrum used for subtraction with initials, date, instrument serial number, identification and supplier/lot number.

7.3.8.4. Record the spectra in the case record

7.3.9. The spectrum may be compared to primary and secondary reference material and / or library searched / compared to aid in interpretation.

7.3.10. The IR spectrum of a substance must be compared to primary or secondary reference material and found to be substantially comparable, i.e., equivalent, to the IR spectrum of the reference material in both its overall appearance and in the presence of major peaks to be considered a positive Category A technique in the identification of a controlled substance, refer to the Drug Chemistry Technical Procedure for Drug Chemistry Analysis.

7.3.10.1. The reference material spectrum must be marked with initials, date, case number, identity, supplier and lot number and included in the case record.

7.3.11. When using IR to determine the salt form of a controlled substance, the areas of the spectrum necessary to identify the salt form must be compared to primary or secondary reference material and found to be substantially comparable, i.e., equivalent, to the IR spectrum of the reference material in both its overall appearance and in the presence of major peaks.

7.3.11.1. The reference material spectrum must be marked with initials, date, case number, identity, supplier and lot number and included in the case record.

7.3.12. The Drug Chemistry Unit Technical Procedure for Extractions may be used to isolate substances from mixtures.

7.3.13. Record the results on the appropriate Drug Chemistry Worksheet form.

7.3.14. Record the date, initials, sample identification, and any comments on the IR activity log.

8. Limitations

8.1. Compounds may exist in different crystal forms, polymorphs, which may produce unique spectra, e.g., mannitol.

8.2. Due caution shall be exercised when using the search or compare function of the instrument software. The Chemist shall evaluate the data and not rely on the computer software for identification.

9. Safety

9.1. Refer to the CCBI Laboratory Safety Manual.

9.2. Do not perform any maintenance or alteration to the instrument that is not described in this procedure. Placing an eye directly in the beam path will cause injury to the eye.

10. References

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10.7. *Instrument Performance Validation Kit User's Guide,* Perkin-Elmer, Inc., UK: 2011.

10.8. *Universal ATR Sampling Accessory User's Guide,* Perkin-Elmer, Inc., UK: 2011.

11. Records

11.1. IR Maintenance Log
11.2. IR Daily QCC Log
11.3. IR Activity Log
11.4. Case record

Revision History		
Effective Date	Version Number	Reason
1/1/13	1	ISO Compliance
2/16/15	2	Updated cleaning instructions in 7.3.1

10: Technical Procedure for Extractions

1. **Purpose / Scope** - This procedure provides direction for the extraction techniques used in the Drug Chemistry Unit of the Raleigh/Wake City-County Bureau of Identification.

2. **Definitions**

   2.1. Quality control check - Periodic confirmation of the reliability of equipment, instrumentation, and/or reagents.
   2.2. Reference Material – Material sufficiently homogeneous and stable with reference to specified properties, which has been established to be fit for its intended use in measurement or in examination of nominal properties

3. **Abbreviations**

   3.1. Refer to the Drug Chemistry Unit Technical Procedure for Drug Chemistry Analysis
   3.2. QCC – Quality control check
   3.3. PQCC – Positive quality control check
   3.4. EXT – extraction
   3.5. "+" or "↑" – used to indicate a fraction collected
   3.6. "-" or "↓" – used to indicate a fraction disposed
   3.7. "→" – used to indicate movement to the next step
   3.8. Evap – evaporated
   3.9. Δ – used to indicate heat
   3.10. X – recrystallize
   3.11. Amm - ammoniated

4. **Materials and Reagents**

   4.1. Materials

   4.1.1. Heat source
   4.1.2. Beakers, vials, test tubes
   4.1.3. Vortex mixer
   4.1.4. Filter Paper
   4.1.5. Funnel
   4.1.6. Glass stirring rod
   4.1.7. Graduated cylinder
   4.1.8. Mortar and pestle
   4.1.9. Pipettes with bulb
   4.1.10. Bottles
   4.1.11. pH Test paper
   4.1.12. Litmus paper

4.1.13. Separatory funnel (optional)
4.1.14. Spatula
4.1.15. Water (Deionized)

4.2. Commercial Reagents

4.2.1. Acids, ACS grade

4.2.1.1. Hydrochloric acid
4.2.1.2. Sulfuric acid
4.2.1.3. Glacial acetic acid

4.2.2. Organic solvents - ACS, Optima or GC Resolv grade

4.2.2.1. Methanol
4.2.2.2. Chloroform
4.2.2.3. Acetone
4.2.2.4. Hexanes
4.2.2.5. Diethyl ether
4.2.2.6. Petroleum ether
4.2.2.7. Ethyl Acetate
4.2.2.8. Isopropanol
4.2.2.9. n-Heptane
4.2.2.10. Ethanol
4.2.2.11. Methylene chloride
4.2.2.12. Cyclohexane

4.2.3. Bases, ACS grade

4.2.3.1. Sodium hydroxide pellets
4.2.3.2. Sodium bicarbonate
4.2.3.3. Ammonium hydroxide

4.2.4. Drying agents, ACS or certified grade

4.2.4.1. Sodium sulfate, anhydrous
4.2.4.2. Magnesium sulfate, anhydrous

4.3. Prepared Reagents - Reagents may be prepared in any amount provided that the component ratios are kept constant. Reagents shall be labeled and stored according to the Drug Chemistry Unit Technical Procedure for Receipt and Quality Assurance of Laboratory Supplies, Reagents, Reference Collections, Reference Standards and Reference Materials.

4.3.1. Dilutions or preparations of acids and bases may be prepared in the molarity or normality desired. Use the molarity or normality and the identity of the acid or base in the lot number.

Label the container clearly with the identity of the acid or base and the molarity or normality.

Example: 5 ml of concentrated HCl is added to 95 ml of water
The initial normality of the HCl is 12N
The final normality is determined: (5 ml)(12 N) / (100 ml) = 0.6 N HCl
The lot number is 20120101HCl0.6NXXX
The container is clearly labeled: 0.6 N HCl

Example: 5 grams of sodium hydroxide pellets are dissolved in 100 ml of water
The formula weight of sodium hydroxide is 39.997
The normality (molarity) is determined:
(5 g / 0.1 L) (1 mol / 39.997 g) = 1.25 N NaOH
The lot number is 20120101NaOH1.25NXXX
The container is clearly labeled: 1.25 N NaOH

4.3.2. Storage: closed container.

4.3.3. Expiration: Stock container: Three years
Use container: One year

4.3.4. Lot number: Eight digit format year/month/day/identity/concentration/initials of preparer.

4.3.5.PQCC: Acceptable result: acidic or basic to litmus or pH paper

4.4. Ammoniated Organic Solvents

4.4.1. Preparation

4.4.1.1. Shake 10 milliliters ammonium hydroxide with 100 milliliters of organic solvent, e.g., hexane, chloroform or a prepared solvent mixture, e.g., 4:1 chloroform:hexane.

4.4.1.2. Allow layers to separate and draw off organic solvent for use.

4.4.2. Storage: closed glass container.

4.4.3. Expiration: stock container: One month
use container: One month

4.4.4. Lot number: eight digit format year/month/day/Amm/solvent identification (ratio) /initials of preparer.
Example: 20120101AmmHexXXX

4.4.5.PQCC: Acceptable result: basic to litmus or pH paper

4.5. Acidified Organic Solvents

4.5.1. Preparation

4.5.1.1. Shake 10 milliliters concentrated hydrochloric acid with 100 milliliters of organic solvent, e.g., diethyl ether, chloroform or a prepared solvent mixture, e.g., 4:1 diethyl ether:hexane.

4.5.1.2. Allow layers to separate and draw off organic solvent for use.

4.5.2. Storage: closed glass container.

4.5.3. Expiration: stock container: One month
use container: One month

4.5.4. Lot number: eight digit format year/month/day/ Acidic/solvent identification (ratio) /initials of preparer.
Example: 20120101AcidicHexXXX

4.5.5. PQCC: Acceptable result: acidic to litmus or pH paper

4.6. Organic solvent mixture

4.6.1. Preparation

4.6.1.1. Mix desired solvents in ratio desired. Mix prior to each use.

4.6.2. Storage: closed glass container.

4.6.3. Expiration: Stock container: Three years
Use container: One year

4.6.4. Lot number: Eight digit format year/month/day/solvent identification and ratio/initials of preparer.
Example: 20120101CHCl3IPA3:1XXX

4.6.5. PQCC: Observe for complete mixing.
Acceptable result: Only one layer is present.

4.7. Procedure

4.7.1. Samples may be extracted to isolate the compound of interest.

4.7.2. Consider the chemical properties, e.g., solubility, partition coefficient and dissociation constant, of the sample and the medium, e.g., pH, and determine the extraction technique. Consider the stability and volatility, especially when applying heat and/or strong acids or bases. Typically basic and acidic drugs are extracted at a pH 2 to 3 units above and below, respectively, the pK_a values of the drugs.

4.7.2.1. Example for an organic basic drug:

- Dissolve the sample in a dilute acid reagent and verify acidity with litmus or pH paper.
- Wash the aqueous solution with an organic solvent chosen based upon the solubilities of the sample and medium to aid in the removal of any unwanted compounds in the medium.
- Repeat the wash if necessary.
- Add a basic reagent to the aqueous solution and verify basicity with litmus or pH paper.
- Wash the aqueous solution with an organic solvent chosen based upon the solubilities of the sample and medium to aid in the removal of any unwanted compounds in the medium.
- Evaporate the solvent in the fume hood, apply heat if desired and the compound of interest is compatible.
    - If excess moisture is a concern, dry the organic solvent over a drying agent, e.g., sodium or magnesium sulfate.
    - If the compound of interest is volatile, add an acidified organic solvent prior to evaporation or evaporate without heat.
- Add a few drops of organic solvent if recrystallization is desired. Apply heat if desired and the compound of interest is compatible.

4.7.2.2. Example for cocaine hydrochloride and inositol:

- Place sample in filter paper over beaker.
- Wash sample in filter paper with chloroform and collect chloroform.
- Evaporate chloroform.

4.7.2.3. Example for hydrocodone and acetaminophen pharmaceutical tablet:

- Crush tablet and place in filter paper.
- Wash sample with diethyl ether and allow material in filter paper to dry.
- Wash sample in filter paper with ammoniated hexane and collect ammoniated hexane.
- Evaporate ammoniated hexane.

4.7.2.4. Example for low dosage pharmaceutical tablet:

- Soak tablet in organic solvent.
- Draw off solvent and filter for GC/MS analysis, refer to the Drug Chemistry Unit Technical Procedure for Gas Chromatography / Mass Spectrometry.

4.7.3. Record the extraction technique used in sufficient detail to allow the technique to be repeated. Record the lot number of any prepared reagents used. When a specific target pH is desired check the pH with pH test paper and record the observed pH.

4.7.4. Negative Control for GC/MS extractions

4.7.4.1. Each extraction analyzed by GC/MS shall be accompanied by a negative control extraction performed immediately prior to or concurrent with the sample extraction using the same techniques, reagents and materials as the sample extraction in approximately the same amounts.

4.7.4.1.1. When using disposable vessels the negative control extraction may be performed in separate glassware from the sample extraction.

4.7.4.1.2. When using reusable vessels, perform the negative control extraction in the vessel immediately prior to performing the sample extraction.

4.7.4.1.3. When filtering, use the filter medium from the negative control extraction for the sample extraction. Do not obtain new filter medium for the sample extraction.

5. Limitations

5.1. Solvents and pH's must be chosen as directed in 4.7.2.

6. Safety – Refer to the CCBI Crime Laboratory Safety Manual

7. Records

7.1. Drug Chemistry worksheets
7.2. Reagent log

8. References

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Revision History		
Effective Date	Version Number	Reason
1/1/13	1	ISO Compliance
2/16/15	2	Added solvents to section 4.2.2 and negative control requirements for GC/MS analysis to section 4.7.4

11: Technical Procedure for Microcrystalline Tests

1. **Purpose / Scope** - This procedure provides instruction for the performance of microcrystalline tests in the Drug Chemistry Unit of the Raleigh/Wake City-County Bureau of Identification.

2. Definitions

2.1. Quality control check - Periodic confirmation of the reliability of equipment, instrumentation, and/or reagents.

2.2. Reference Material – Material sufficiently homogeneous and stable with reference to specified properties, which has been established to be fit for its intended use in measurement or in examination of nominal properties.

3. Abbreviations

3.1. Refer to the Drug Chemistry Unit Technical Procedure for Drug Chemistry Analysis

4. Equipment, Materials and Reagents

4.1. Equipment

4.1.1. Nikon Eclipse E400 Pol polarizing microscope equipped with 10X eyepiece and 10X objective to produce magnification of 100X
4.1.2. Balance

4.2. Materials

4.2.1. Beakers or other glass vessels
4.2.2. Graduated cylinder
4.2.3. Glass stirring rod
4.2.4. Reagent bottle(s)
4.2.5. Microscope slides
4.2.6. Spatula
4.2.7. Weigh boats or other weigh vessels
4.2.8. Water

4.3. Reference Material

4.3.1. Cocaine
4.3.2. Heroin
4.3.3. Caffeine

4.4. Reagents

4.4.1. Mercuric chloride, ACS grade
4.4.2. Gold chloride trihydrate, ACS grade
4.4.3. Glacial acetic acid, ACS grade

5. Standards and Controls

5.1. Reagents shall be prepared, labeled and stored in accordance with the Drug Chemistry Unit Technical Procedure for Receipt and Quality Assurance of Laboratory Supplies, Reagents, Reference Collections, Reference Standards and Reference Materials.

5.2. Perform positive and negative quality control checks on all Use Containers of microcrystalline test reagents prior to use for analysis. The quality control checks must have acceptable results prior to use of the reagent for analysis. Refer to the CCBI Crime Laboratory Administrative Procedure for Corrective and Preventive Action if necessary.

5.3. Perform negative quality control checks (NQCC) according to the procedure with no sample present.

5.3.1. Acceptable result is no crystal formation, i.e., Negative.

5.3.2. If crystals do form, steps will be taken until no crystals are formed. These steps may include retesting with a new microscope slide, re-cleaning any utensils used, or making a new reagent.

5.3.3. Record any observations and the results of each quality control check on the prepared reagent log.

5.4. Perform positive quality control checks (PQCC) according to the using the specified reference material.

5.4.1. Refer to each microcrystalline test for acceptable results.

5.4.1.1. If acceptable results are not observed, steps will be taken until acceptable results are obtained. These steps may include retesting with a new microscope slide, re-cleaning any utensils used, or making a new reagent.

5.4.1.2. Record any observations and the results of each quality control check on the prepared reagent log.

6. Operation of the Polarizing Microscope

6.1. Refer to the Drug Chemistry Unit Technical Procedure for General Laboratory Equipment.

6.1.1. Turn on the light source.

6.1.2. Place the specimen slide on the stage.

6.1.3. Adjust the desired light intensity with the control lever.

6.1.4. Make sure the field diaphragm is open to the edge of the field view.

6.1.5. Focus with the coarse and fine adjustments for the desired objective.

6.1.6. Move the microscope slide around to view the entire specimen, adjusting the focus accordingly.

6.1.7. Push the filter in to view the specimen with polars crossed, or pull it out to view with uncrossed polars.

7. Procedure

7.1. Place a small portion of the sample, a few particles of material, on a microscope slide and add a drop of the microcrystalline reagent (A), refer to section 8, and mix with the sample.

7.2. If dilution is necessary, mix the sample with a drop of reagent (B), refer to section 8, on the microscope slide prior to adding reagent (A), refer to section 8.

7.3. Samples that have evaporated to dryness shall not be used for evaluation of crystals.

7.4. Observe any crystals on the polarizing microscope under non-polarized and/or polarized light.

7.4.1. If no crystals are observed record on the Drug Chemistry Worksheet form.

7.5. Crystals shall be compared morphologically to those of reference materials tested contemporaneously. If the crystal observed is that of a reference material used for a PQCC recorded by the Drug Chemist performing the microcrystalline test then the reference material need not be analyzed contemporaneously with the sample.

7.5.1. A positive comparison occurs when the observed sample crystal is morphologically the same as the observed reference material crystal.

7.5.1.1. Record a description, drawn and / or described in words, of the crystals observed for the sample and the reference material, if applicable, on the Drug Chemistry Worksheet form and the identity, supplier and lot number of the reference material.

7.5.2. If the observed sample crystal and the observed reference material crystal are not morphologically the same then the comparison is negative.

7.5.2.1. Record a description, drawn and / or described in words, of the crystals observed for the sample and the reference material, if applicable, on the Drug Chemistry Worksheet form and the identity, supplier and lot number of the reference material.

8. Microcrystalline Reagents

8.1. 5 % Mercuric Chloride

8.1.1. This reagent is used for heroin and caffeine.

8.1.2. Selected characteristic results:

8.1.2.1. Heroin – fans / dendrites
8.1.2.2. Caffeine – dendrites which are longer and less dense than heroin dendrites

8.1.3. Preparation

8.1.3.1. 5 % Mercuric Chloride, w/v (A)

8.1.3.1.1. Dissolve 1.5 grams of mercuric chloride in 30 milliliters of water

8.1.3.1.2. Storage: closed container

8.1.3.1.3. Expiration: Stock container: Three years
Use container: One year

8.1.3.1.4. Lot number: Eight digit format year/month/day/HgCl2/Initials of preparer. Example: 20101231HgCl2XXX

8.1.3.1.5. PQCC

8.1.3.1.5.1. Reference material: Heroin
8.1.3.1.5.2. Acceptable result: Fans / dendrites

8.1.3.2. 0.05 N Hydrochloric Acid (B)

8.1.3.2.1. Mix 1 milliliter of concentrated hydrochloric acid with 250 milliliters of deionized water.

8.1.3.2.2. Storage: closed container

8.1.3.2.3. Expiration: Stock container: Three years
Use container: One year

8.1.3.2.4. Lot number: Eight digit format year/month/day/HCl0.05N/Initials of preparer. Example: 20101231HCl0.05NXXX

8.1.3.2.5. PQCC

8.1.3.2.5.1. Reference material: Heroin
8.1.3.2.5.2. Acceptable result: Fans / dendrites

8.2. Gold Chloride in 20 % Acetic Acid

8.2.1. This reagent is used for cocaine.

8.2.2. Selected characteristic results:

8.2.2.1. Cocaine - cross-shaped crystals.

8.2.3. Preparation

8.2.3.1. 2% Gold Chloride (w/v) in 20 % Acetic Acid (v/v) (A)

8.2.3.1.1. Add 10 milliliters glacial acetic acid to 40 milliliters of water, mix.

8.2.3.1.2. Dissolve 1.0 gram of gold chloride in the 50 milliliters of 20 % acetic acid, with stirring.

8.2.3.1.3. Storage: Closed container

8.2.3.1.4. Expiration: Stock container: Three years
Use container: One year

8.2.3.1.5. Lot number: Eight digit format year/month/day/AuCl/Initials of preparer. Example: 2010123AuClXXX

8.2.3.1.6. PQCC

8.2.3.1.6.1. Reference material: Cocaine
8.2.3.1.6.2. Acceptable result: Cross-shaped crystals

8.2.3.2. 0.05 N Hydrochloric Acid (B)

8.2.3.2.1. Refer to 8.1.3.2.

9. Limitations

9.1. Diluents and adulterants may interfere with crystal formation. Refer to the Drug Chemistry Unit Technical Procedure for Extractions to remove unwanted components prior to microcrystalline testing.

9.2. Concentration of samples may need to be increased or decreased to aid in crystal formation.

10. Safety – Refer to the CCBI Crime Laboratory Safety Manual

11. References

11.1. *Nikon Polarizing Microscope Eclipse E400Pol Instructions*, Nikon Inc, Melville, NY, M216E 98.8.VF.1.

11.2. Clarke, E.G.C., and R.G. Todd, eds. *Isolation and Identification of Drugs*. 1st Edition. London: Pharmaceutical Press, 1969: 135-141, 801.

11.3. Allen, A. C., Copper, D. A., Kiser, W. O., Cottrell, R. C., "The Cocaine Diastereoisomers," *Journal of Forensic Sciences*, Vol. 26, No.1, Jan. 1981, pp. 12–26.

11.4. Smith, F.P., ed. *Handbook of Forensic Drug Analysis.* Boston, Massachusetts: Elsevier Academic Press, 2005: 238.

12. Records

12.1. Reagent log
12.2. Case record

Revision History		
Effective Date	Version Number	Reason
1/15/13	1	ISO Compliance

12: Gas Chromatography/Mass Spectrometry (GC-MS)

1. **Purpose / Scope** - This procedure provides direction for the initial setup, performance checks and usage of gas chromatograph – mass spectrometer instruments in the Drug Chemistry Unit of the Raleigh/Wake City-County Bureau of Identification.

2. **Definitions**

   2.1. Performance verification - The initial confirmation of the reliability of a previously or externally validated method or instrument.
   2.2. Quality control check - Periodic confirmation of the reliability of equipment, instrumentation, and/or reagents.
   2.3. Reference Material – Material sufficiently homogeneous and stable with reference to specified properties, which has been established to be fit for its intended use in measurement or in examination of nominal properties

3. **Abbreviations**

   3.1. Refer to the Drug Chemistry Unit Technical Procedure for Drug Chemistry Analysis
   3.2. QCC – Quality Control Check
   3.3. GC – Gas chromatograph
   3.4. MS – Mass spectrometer
   3.5. MSD - Mass Selective Detector
   3.6. TIC – Total ion chromatogram
   3.7. RT – Retention time

4. **Equipment, Materials and Reagents**

   4.1. Equipment

      4.1.1. Agilent 6890 Gas Chromatograph with Agilent 5975 Series Mass Selective Detector with Agilent Automatic Liquid Sampler and tray
      4.1.2. Computer with Agilent Analytical MSD Productivity ChemStation Software and Printer

   4.2. Reference Materials

      4.2.1. Multi-component drug solution containing alprazolam, clonazepam, diazepam, flunitrazepam, lorazepam, nitrazepam, oxazepam and temazepam

   4.3. Materials

      4.3.1. Sample vials, caps and inserts
      4.3.2. ALS Syringe, 10μl straight, fixed needle, 23/42/HP

4.3.3. DB5-MS Column, 30 m X 0.250 mm X 0.25 µm
4.3.4. Agilent inlet liner, split, single taper with glass wool, deactivated
4.3.5. Agilent liner O-ring, non-stick flip-top
4.3.6. Non-stick septa, 11mm
4.3.7. Septum wrench
4.3.8. Tweezers
4.3.9. Clean, lint free, non-nylon gloves
4.3.10. Wrenches, ¼ inch and ½ inch
4.3.11. Gold plated inlet seal with cross and 0.375 outer diameter washer
4.3.12. Star or Torx screwdriver
4.3.13. Flat head screwdriver, large
4.3.14. Hex key, 5 mm
4.3.15. Inland 45 pump oil
4.3.16. Funnel
4.3.17. Hex ball driver, 1.5 mm
4.3.18. Hex ball driver, 2.0 mm
4.3.19. Wrench, open-end, 10 mm
4.3.20. Alumina abrasive powder
4.3.21. Cotton swabs
4.3.22. Ultrasonic bath

4.4. Commercial Reagents

4.4.1. Methanol, Optima or GC Resolv grade
4.4.2. Hexanes, Optima grade
4.4.3. Chloroform, Optima grade
4.4.4. Acetonitrile, Optima grade
4.4.5. Ethyl acetate, Optima grade
4.4.6. Methylene chloride, Optima or Pesticide grade
4.4.7. Helium gas, Grade 5.0
4.4.8. Perfluorotributylamine [PFTBA], neat

5. Standards and Controls

5.1. A GC-MS logbook shall be maintained near the instrument. The logbook shall contain the GC-MS Activity Log, GC-MS Daily QCC Log, the GC-MS Monthly QCC Log, the GC-MS Maintenance Logs and any manufacturer's certificates, performance verification documentation, monthly and daily QCC printouts, printed sequence logs and maintenance documentation.

5.1.1. The logbook shall contain the Drug Chemistry GC-MS activity log.

5.1.1.1. Record the date, sample identification, initials of operator, GC-MS method used, any comments and substances observed in the sample for each sample analyzed on the Drug Chemistry GC-MS Activity Log.

5.1.1.1.1. If samples are rerun for any reason, record a new entry on the Drug Chemistry GC-MS Activity Log.

5.1.1.2. Record any error messages on the Drug Chemistry GC-MS Activity Log.

5.1.2. The logbook shall contain the Drug Chemistry GC-MS daily QCC log.

5.1.2.1. Record all Daily QCC's on the Daily QCC log with the date, initials, and results and any comments.

5.1.2.2. Place Daily QCC printouts in the instrument logbook.

5.1.3. The logbook shall contain the monthly QCC data, refer to 5.7. Other reference material retention time data may be maintained in the logbook.

5.1.4. The logbook shall contain the Drug Chemistry GC-MS maintenance logs, refer to Section 6.

5.1.5. Archive the instrument logbook yearly.

5.1.5.1. Label the instrument logbook with the instrument serial number and year and store near the instrument.

5.2. When the GC-MS has been placed out of service for non-routine maintenance, malfunction or leaving direct control of the Laboratory the Drug Chemistry Technical Leader shall evaluate the instrument and determine if any additional quality control checks are needed to ensure instrument performance. At a minimum, a Daily QCC must be successfully performed prior to placing the instrument back in service, refer to 5.6.

5.2.1. If maintenance is performed that may affect retention times, a monthly QCC, refer to 5.7., shall be performed before the instrument is placed back in service.

5.3. The Drug Chemist shall record any malfunctions or error messages in the GC-MS Activity Log, notify the Drug Chemistry Technical Leader of any malfunctions or error messages and place the instrument out of service by marking the GC-MS Activity Log "Out of Service."

5.4. The Drug Chemistry Technical Leader shall correct any problems with the instrument or request service. The Drug Chemistry Technical Leader shall examine the effect(s), if any, of a malfunction or error message on analysis results and implement the CCBI Laboratory Procedure for Corrective and Preventive Action as required.

5.5. Negative Quality Control Check

5.5.1. Negative QCC's are performed prior to each sample injection, refer to 7.3.

5.6. Daily Quality Control Check – Standard Spectra Tune

5.6.1. Perform a Standard Spectra Tune (stune) with Perfluorotributylamine (PFTBA) as the tuning standard prior to beginning the first sample sequence each day the instrument is in use.

5.6.1.1. Sample sequences that continue overnight may be allowed to complete without performing a new tune provided that they do not extend more than twenty-four hours beyond the time of the tune or noon, whichever is later.

5.6.2. Compare the stune report to previous ones. Record any major variations on the stune report and on the Daily QCC log and notify the Drug Chemistry Technical Leader.

5.6.3. The mass assignments of the three tuning masses in the upper part of the report must be within +/- 0.2 amu of 69.00, 219.00, and 502.00.

5.6.4. The peak widths of the three tuning masses must be within +/- 0.10 amu of 0.55 and the peaks must generally be smooth and symmetrical.

5.6.5. The base peak must be identified as mass 69. The relative abundance ratio of mass 219 to mass 69 must be within 40 – 85 % and the relative abundance ratio of mass 502 to mass 69 must be within 2.0 – 5 %.

5.6.6. The 70/69 isotopic ratio must be from 0.5 – 1.6, the 220/219 ratio must be from 3.2 – 5.4, and the 503/502 the ratio must be from 7.9 – 12.3.

5.6.7. If any parameter listed in 5.6.3. - 5.6.6. do not meet the requirements listed, document the deviation on the stune report and on the GC-MS daily QCC log.

5.6.7.1. Perform another stune.

5.6.7.2. If the problem persists, place the instrument out of service by marking the activity log “out of service” and notify the Drug Chemistry Technical Leader.

5.6.7.3. The Drug Chemistry Technical Leader shall correct any problems with the instrument or request service. The Drug Chemistry Technical Leader shall examine the effect(s), if any, on analysis results and implement the CCBI Laboratory Procedure for Corrective and Preventive Action as required.

5.6.7.4. The daily QCC must be successfully completed prior to placing the instrument back in service.

5.6.8. The abundance of any peaks less than 69 amu must not be greater than 10 % of the abundance of the base peak.

5.6.8.1. Peaks at 18, 28 or 32 amu are indicative of water, nitrogen and oxygen, respectively, and may indicate an air leak. Other peaks may indicate gas impurities.

5.6.8.2. If an air leak is detected, isolate the leak and tighten fittings to correct the leak and perform another stune.

5.6.8.3. If the problem persists, place the instrument out of service by marking the activity log "out of service" and notify the Drug Chemistry Technical Leader.

5.6.8.4. The Drug Chemistry Technical Leader shall correct any problems with the instrument or request service. The Drug Chemistry Technical Leader shall examine the effect(s), if any, on analysis results and implement the CCBI Laboratory Procedure for Corrective and Preventive Action as required.

5.6.8.5. The daily QCC must be successfully completed prior to placing the instrument back in service.

5.6.9. Initial the stune report and mark any parameter that does not meet the requirement specified. Place the stune report in the instrument logbook and record the results in the GC-MS daily QCC log.

5.7. Monthly Quality Control Check

5.7.1. The multi-component drug solution from 4.2.1. shall be injected on each method, refer to 7.1., each month the instrument is in use to verify instrument performance.

5.7.2. The solution shall, when feasible, be run during the first seven calendar days of each month.

5.7.2.1. If the standard solution is not run during the first seven calendar days of the month, the instrument shall be out of service until the standard solution is successfully run.

5.7.3. Name the multi-component reference material solution data files with "MC" followed by the numerical year and month designation and the method name. Name the corresponding solvent blank with the same designation followed by "-b", indicating that it is a blank.

Example: The name of a standard solution run in January, 2099 on the LOW method would be "MC209901LOW" and the blank would be "MC209901LOW-b."

5.7.4. Visually examine the TIC of the monthly QCC solution for chromatographic quality and resolution. All components must exhibit visually symmetrical peaks that are visually baseline resolved or for peaks separated by 0.2 minutes or less, visually resolved at half height.

5.7.5. Perform a GC retention time comparison, refer to 7.10., for the Oxazepam, Temazepam and Alprazolam components of the monthly QCC solution to those of the previous monthly QCC solution run.

5.7.6. Perform a mass spectral comparison, refer to 7.9., for the Oxazepam, Temazepam and Alprazolam components of the monthly QCC solution to reference material.

5.7.7. Each component must have a positive comparison for 5.7.5. and 5.7.6.

5.7.7.1. Record any component that does have a positive comparison on the instrument monthly QCC log, place the instrument out of service by marking the GC-MS activity log "out of service" and notify the Drug Chemistry Technical Leader.

5.7.7.2. The Drug Chemistry Technical Leader shall correct any problems with the instrument or request service. The Drug Chemistry Technical Leader shall examine the effect(s), if any, on analysis results and implement the CCBI Laboratory Procedure for Corrective and Preventive Action as required.

5.7.7.3. The monthly QCC must be successfully completed prior to placing the instrument back in service.

5.7.8. Print the monthly QCC TIC with the retention times displayed, the mass spectrum of each component and the corresponding blank TIC.

5.7.8.1. Mark each page with initials and date and note any problems.

5.7.8.2. Record the lot number ~~and supplier~~ of the standard solution on the TIC.

5.7.8.3. File the generated data in the instrument logbook.

5.7.8.4. Record the monthly check in the monthly QCC log.

5.7.9. Additional reference material solutions may be run on a monthly basis to establish retention times.

5.8. Performance Verification for New Instrumentation

5.8.1. New GC-MS instruments shall be installed by a manufacturer representative or approved vendor according to the manufacturer's guidelines.

5.8.2. Prior to use, perform daily QCC's, refer to 5.6., on three separate days. The daily QCC's must meet all specified requirements.

5.8.3. Prior to use, analyze the multi-component drug solution from 4.2.1 on each method (refer to 7.1.) on three separate days.

5.8.3.1. The mass spectra of each component must have a positive mass spectral comparison to reference material, refer to 7.9.

5.8.3.2. The retention times of each component must have a positive GC retention time comparison to reference material, refer to 7.10.

5.8.4. Label the instrument printouts with the lot number of the reference material, initials and date. Record the performance verification in the GC-MS logbook and place the printouts in the GC-MS logbook.

5.8.5. The performance verification must be reviewed and approved by the Drug Chemistry Technical Leader prior to the instrument being used for casework. The Drug Chemistry Technical Leader shall record the review and approval in the GC-MS logbook.

6. Maintenance

6.1. Place the instrument out of service prior to performing any maintenance, other than wash vial and syringe maintenance, by marking the GC-MS activity log "Out of Service." When the instrument is ready to be returned to service, mark the GC-MS activity log "Back in Service."

6.2. Record all maintenance one of the GC-MS maintenance logs at the time it is performed with the name of the person performing the maintenance or repairs, the initials of the Drug Chemist recording the maintenance or repairs, the date, a description of the maintenance or repairs and a list of any parts replaced.

6.3. Record lengths of column trimmed in the activity log and a maintenance log. If the column is trimmed, the instrument shall be out of service until a monthly QCC is successfully completed, refer to 5.7.

6.3.1. Reference materials ran prior to the column maintenance shall not be used for retention time comparison after the column maintenance.

6.3.2. The Drug Chemistry Technical Leader shall update the instrument log when the instrument is ready to be used for casework and file any generated data in the instrument logbook located near the instrument.

6.4. **Suggested Routine maintenance** – The routine maintenance schedule is a suggested guideline. The maintenance schedule will be determined by the Drug Chemistry Technical Leader based upon instrument usage and performance.

6.4.1. Wash Vials

6.4.1.1. Rinse and fill daily when in use. Use methanol in the first wash vial and ethyl acetate in the second wash vial.

6.4.1.2. Record on the GCMS Daily Maintenance Log.
6.4.1.3. Required post-maintenance check: None.

6.4.2. Septum

6.4.2.1. Replace weekly, at a minimum, when in use.

6.4.2.1.1. Press [Oven] and set the oven to 35°C. When the temperature reaches setpoint, turn the oven off. Press [Front Inlet] and turn off the inlet temperature and pressure.
6.4.2.1.2. Be careful - The inlet fittings may be hot enough to cause burns. Remove the septum retainer nut, using the wrench if the nut is hot or sticks.
6.4.2.1.3. Remove the old septum, using tweezers if necessary. Be sure that it is completely removed and take care to avoid gouging or scratching the interior of the septum head.
6.4.2.1.4. Press a new septum into place firmly.
6.4.2.1.5. Replace the septum retainer nut, tightening it finger-tight until the c-ring is about 1 mm above the nut. Avoid overtightening.
6.4.2.1.6. Using ChemStation load a method to return the GC to appropriate settings. If prompted, do not save any method changes.
6.4.2.1.7. Allow the GC to return to the setpoints.

6.4.2.2. Record on the GCMS Weekly Maintenance Log.
6.4.2.3. Required post-maintenance check: Successful daily QCC, refer to 5.6.

6.4.3. Syringe

6.4.3.1. Inspect monthly for cleanliness and ease of movement. Replace as needed.

6.4.3.1.1. Mount the injector on a parking post.
6.4.3.1.2. Open the injector door.
6.4.3.1.3. Slide the syringe carriage to the top position.
6.4.3.1.4. Completely loosen the plunger thumb screw until it reaches the stop, and lift the plunger carrier off of the syringe plunger.
6.4.3.1.5. Open the syringe latch by swinging it in a counterclockwise direction.
6.4.3.1.6. Carefully pull the top of the syringe out of the flange guide, then lift the needle out of the needle support foot.
6.4.3.1.7. Carefully pass the new syringe needle through the guide hole in the needle support foot.
6.4.3.1.8. Align the syringe flange with the flange guide and press the syringe into place, keeping the needle end in the guide hole of the needle support foot. Make sure that the flat edge of the syringe flange faces out.
6.4.3.1.9. Close the syringe latch by swinging it clockwise until it snaps in place.
6.4.3.1.10. Slide the plunger carrier down until it is completely over the syringe plunger, and tighten the plunger thumb screw until finger- tight.

6.4.3.1.11. Manually move the plunger carrier up and down. If the syringe plunger does not move along with the carrier, repeat the previous steps until installed correctly. Be sure the plunger thumb screw is secure and tight. Verify that the needle is inside the guide hole of the needle support foot. The needle should be straight and pass freely through the needle guide hole. If the needle is bent or is outside the guide hole, remove the syringe and reinstall.

6.4.3.1.12. Close the injector door.

6.4.3.1.13. Mount the injector on the inlet.

6.4.3.2. Record on the GCMS Monthly Maintenance Log.

6.4.3.3. Required post-maintenance check: None.

6.4.4. Liner

6.4.4.1. Replace monthly, at a minimum, when in use.

6.4.4.1.1. Press [Oven] and set the oven to 35°C. When the temperature reaches setpoint, turn the oven off. Press [Front Inlet] and turn off the inlet temperature and pressure.

6.4.4.1.2. Be careful - The inlet fittings may be hot enough to cause burns. Flip the inlet open.

6.4.4.1.3. Remove liner with tweezers, being careful not to break the liner.

6.4.4.1.4. Hold the new liner with tweezers or lint free tissue and place the o-ring on the liner about 2 to 3 mm from its top end.

6.4.4.1.5. Insert the liner straight down into the inlet and press gently to ensure it is seated.

6.4.4.1.6. Replace the inlet cover and flip the top into place.

6.4.4.1.7. Using ChemStation load a method to return the GC to appropriate settings. If prompted, do not save any method changes.

6.4.4.1.8. Allow the GC to return to the setpoints.

6.4.4.2. Record on the GCMS Monthly Maintenance Log.

6.4.4.3. Required post-maintenance check: Successful daily QCC, refer to 5.6.

6.4.5. Pump Oil

6.4.5.1. Change every six months.

6.4.5.1.1. Vent the MSD by selecting the vent option in Instrument Control of Chemstation. Allow the vent cycle to run, when the cycle is complete and the temperatures are below 100 degrees Celsius turn off the MSD.

6.4.5.1.2. Press [Oven] and set the oven to 35°C. Press [Front Inlet] and turn off the inlet temperature and pressure. When the temperature reaches the setpoint turn the GC off.

6.4.5.1.3. Place a book or other object approximately two inches thick under the pump motor to tilt it up slightly.
6.4.5.1.4. Remove the fill cap.
6.4.5.1.5. Place a container under the drain plug.
6.4.5.1.6. Remove the drain plug. Allow the pump oil to drain out. The foreline pump and oil may be hot.
6.4.5.1.7. Replace the drain plug.
6.4.5.1.8. Remove the book or object used to prop up the pump.
6.4.5.1.9. Refill the foreline pump with Inland 45 pump oil, using a funnel, until the oil level is between the two fill marks in the site window, approximately 0.28 L of oil.
6.4.5.1.10. Wait a few minutes for the oil to settle. If the oil level drops, add oil to bring the oil level near the upper line.
6.4.5.1.11. Reinstall the fill cap.
6.4.5.1.12. Power on the GC.
6.4.5.1.13. Ensure that the vent valve is closed. Holding the MSD chamber door closed, power on the MSD and ensure that the turbo pump speed climbs to 100%.
6.4.5.1.14. Start the Chemstation software and apply setpoints.
6.4.5.1.15. Allow the instrument to equilibrate for two hours prior to tuning.

6.4.5.2. Record on the GCMS Biannual Maintenance Log.
6.4.5.3. Required post-maintenance check: Successful daily QCC, refer to 5.6.

6.4.6. Clean Source

6.4.6.1. Clean annually, at a minimum.

6.4.6.1.1. Vent the MSD by selecting the vent option in Instrument Control of Chemstation. Allow the vent cycle to run, when the cycle is complete and the temperatures are below 100 degrees Celsius turn off the MSD.
6.4.6.1.2. Press [Oven] and set the oven to 35°C. Press [Front Inlet] and turn off the inlet temperature and pressure. When the temperature reaches the setpoint turn the GC off.
6.4.6.1.3. Open the vent valve
6.4.6.1.4. Detach the ribbon cables from the circuit board on the MSD chamber door.
6.4.6.1.5. Pull open the MSD chamber door by hand.
6.4.6.1.6. Detach the leads from the ion source, loosen the screws and remove the ion source.
6.4.6.1.7. Remove the filaments using a hex ball driver.
6.4.6.1.8. Separate the repeller assembly from the source body. The repeller assembly includes the source heater assembly, repeller, and related parts.
6.4.6.1.9. Remove the repeller.

6.4.6.1.10. Unscrew the interface socket. A 10-mm open-end wrench fits the flats on the interface socket.

6.4.6.1.11. Remove the setscrew for the lenses.

6.4.6.1.12. Push the lenses out of the source body.

6.4.6.1.13. If insulators are dirty, clean them with a cotton swab dampened with reagent-grade methanol. If that does not clean the insulators, replace them. Do not abrasively or ultrasonically clean the insulators.

6.4.6.1.14. The filaments and source heater assembly cannot be cleaned ultrasonically. Replace these components if major contamination occurs.

6.4.6.1.15. Collect the following parts that contact the sample or ion beam to be cleaned.

6.4.6.1.15.1. Repeller

6.4.6.1.15.2. Interface socket

6.4.6.1.15.3. Source body

6.4.6.1.15.4. Drawout plate

6.4.6.1.15.5. Drawout cylinder

6.4.6.1.15.6. Ion focus lens

6.4.6.1.15.7. Entrance lens

6.4.6.1.16. Abrasively clean the surfaces that contact the sample or ion beam.

6.4.6.1.17. Use an abrasive slurry of alumina powder and methanol on a cotton swab. Use enough force to remove all discolorations. Polishing the parts is not necessary; small scratches will not harm performance. Also abrasively clean the discolorations where electrons from the filaments enter the source body.

6.4.6.1.18. Rinse away all abrasive residue with reagent-grade methanol.

6.4.6.1.19. Make sure all abrasive residue is rinsed way before ultrasonic cleaning. If the methanol becomes cloudy or contains visible particles, rinse again.

6.4.6.1.20. Separate the parts that were abrasively cleaned from the parts that were not abrasively cleaned.

6.4.6.1.21. Ultrasonically clean the parts (each group separately) for 15 minutes in each of the following solvents: methylene chloride followed by acetone followed by methanol.

6.4.6.1.22. Place the parts in a clean beaker. Loosely cover the beaker with clean aluminum foil (dull side down).

6.4.6.1.23. Dry the cleaned parts in an oven at 100 °C for 5–6 minutes.

6.4.6.1.24. Let the parts cool before you handle them.

6.4.6.1.25. Take care to avoid recontaminating cleaned and dried parts. Put on new, clean gloves before handling the parts. Do not set the cleaned parts on a dirty surface. Set them only on clean, lint-free cloths.

6.4.6.1.26. Slide the drawout plate and the drawout cylinder into the source body.

6.4.6.1.27. Assemble the ion focus lens, entrance lens, and lens insulators.

6.4.6.1.28. Slide the assembled parts into the source body.

6.4.6.1.29. Install the setscrew that holds the lenses in place.

6.4.6.1.30. Reinstall the repeller, repeller insulators, washer, and repeller nut into the source heater assembly. The resulting assembly is called the repeller assembly.

6.4.6.1.31. Reconnect the repeller assembly to the source body. The repeller assembly includes the source heater assembly, repeller, and related parts.
6.4.6.1.32. Reinstall the filaments, replace if excessively worn.
6.4.6.1.33. Reinstall the interface socket.
6.4.6.1.34. Do not overtighten the repeller nut or the ceramic repeller insulators will break when the source heats up. The nut should only be finger-tight.
6.4.6.1.35. Do not overtighten the interface socket. Overtightening could strip the threads.
6.4.6.1.36. Reinstall the ion source into the MSD and reattach the leads.
6.4.6.1.37. Close the vent valve.
6.4.6.1.38. Push the MSD chamber door close and reattach the ribbon cables to the circuit board.
6.4.6.1.39. Power on the GC.
6.4.6.1.40. Holding the MSD chamber door closed, power on the MSD and ensure that the turbo pump speed climbs to 100%.
6.4.6.1.41. Start the Chemstation software and apply setpoints.
6.4.6.1.42. Allow the instrument to equilibrate for two hours prior to tuning.

6.4.6.2. Record on the GCMS Annual Maintenance Log.
6.4.6.3. Required post-maintenance check: Successful daily QCC, refer to 5.6., and monthly QCC, refer to 5.7.

6.4.7. Gold Seal

6.4.7.1. Replace annually, at a minimum.

6.4.7.1.1. Vent the MSD by selecting the vent option in Instrument Control of Chemstation. Allow the vent cycle to run, when the cycle is complete and the temperatures are below 100 degrees Celsius turn off the MSD.
6.4.7.1.2. Press [Oven] and set the oven to 35°C. Press [Front Inlet] and turn off the inlet temperature and pressure. When the temperature reaches the setpoint turn the GC off.
6.4.7.1.3. Be careful - The inlet fittings may be hot enough to cause burns. Loosen the inlet column nut with the ¼ inch wrench and remove the column from the inlet. Cap the open end of the column to prevent contamination.
6.4.7.1.4. Remove the insulation cup from around the base of the inlet using the star screwdriver.
6.4.7.1.5. Use the 1/2-inch wrench to loosen the reducing nut, and then remove it.
6.4.7.1.6. The washer and seal are inside the reducing nut. Remove them, noting their orientation.
6.4.7.1.7. Handle the new gold seal and washer with clean, lint-free, non-nylon gloves. Place the washer in the reducing nut. Place the new inlet base seal on top of it with the raised portion facing down.
6.4.7.1.8. Replace the reducing nut. Use the 1/2-inch wrench to tighten the nut.
6.4.7.1.9. Replace the column and the insulation cup.

6.4.7.1.10. Using ChemStation load a method to return the GC to appropriate settings. If prompted, do not save any method changes.

6.4.7.1.11. Allow the GC to return to the setpoints.

6.4.7.1.12. Ensure that the vent valve is closed. Holding the MSD chamber door closed, power on the MSD and ensure that the turbo pump speed climbs to 100%.

6.4.7.1.13. Start the ChemStation software and apply setpoints.

6.4.7.1.14. Allow the instrument to equilibrate for two hours prior to tuning.

6.4.7.2. Record on the GCMS Annual Maintenance Log.

6.4.7.3. Required post-maintenance check: Successful daily QCC, refer to 5.6., and monthly QCC, refer to 5.7.

7. Procedure

7.1. Instrument Settings

7.1.1. Select a GC-MS method based on the sample and any analysis results.

7.1.1.1. The SCREEN (SCRN) method shall be used when a controlled substance is not previously indicated and a GC-MS analysis is performed, i.e. negative preliminary testing and/or infrared analysis indicated a non-controlled substance and a GC-MS analysis is performed.

7.1.1.2. The SCREEN (SCRN) method shall be used for at least one sample preparation when GC-MS is the sole technique used in analysis.

7.1.1.3. Each method may be used with split ratios of 5:1, 20:1, or 100:1. Numbers in front of the method name indicates the split ratio.

7.1.1.4. Splitless injections are generally not utilized, but may be used for sample preparations that did not provide successful GC or MS comparison of a compound using a 5:1 or higher split ratio. "NoSplit" in front of the method name indicates a splitless injection.

7.1.1.5. Each method shall wash the syringe at least 10 times between injections to ensure sample integrity

7.1.1.6. When the standard methods are not appropriate to analyze a compound, a modified method may be used in accordance with the CCBI Administrative Procedure for Exceptions.

7.1.2. HIGH – typically used for compounds that elute after 13 minutes in the screen method, e.g. buprenorphine, LSD, some steroids and some synthetic cannabinoids.

7.1.2.1. 1.0 minute initial time
7.1.2.2. 280 °C initial temperature
7.1.2.3. 10 °C/minute ramp
7.1.2.4. 300 °C final temperature
7.1.2.5. 22.0 minutes final time
7.1.2.6. 25.0 minutes total time
7.1.2.7. Scan range = 40-500 amu
7.1.2.8. 230 °C source temperature
7.1.2.9. 150 °C quadrupole temperature

7.1.3. LOW – typically used for compounds that elute prior to 13 minutes in the screen method, e.g. cocaine, amphetamines, some steroids, some synthetic cannabinoids, most opiates and most benzodiazepines.

7.1.3.1. 1.5 minutes initial time
7.1.3.2. 100 °C initial temperature
7.1.3.3. 30 °C/minute ramp
7.1.3.4. 300 °C final temperature
7.1.3.5. 4.83 minutes final time
7.1.3.6. 13.0 minutes total time
7.1.3.7. Scan range = 40-500 amu
7.1.3.8. 230 °C source temperature
7.1.3.9. 150 °C quadrupole temperature

7.1.4. SCREEN (SCRN) – Use this method when GC-MS is used to screen samples to determine if a controlled substance may be present.

7.1.4.1. 1.5 minutes initial time
7.1.4.2. 100 °C initial temperature
7.1.4.3. 30 °C/minute
7.1.4.4. 300 °C final temperature
7.1.4.5. 26.83 minutes final time
7.1.4.6. 35.0 minutes total time
7.1.4.7. Scan range = 40-500 amu
7.1.4.8. 230 °C source temperature
7.1.4.9. 150 °C quadrupole temperature

7.2. Shutdown / Startup

7.2.1. The GC-MS shall be left on at all times.

7.2.2. The computer may be shut down or restarted if needed.

7.2.3. A successful daily QCC check, refer to 5.6., must be performed following any GC or MS shutdown.

7.2.4. Record any shutdown on the GC-MS activity.

7.3. Prior to the injection of a sample, perform a ~~blank solvent injection as a~~ negative QCC injection using the negative quality control extraction prepared using the same techniques, reagents materials, reagents and solvents as the sample preparation, refer to DCTP10. Use the same method and split ratio as the sample.

7.3.1. Prepare the negative QCC extraction ~~blank solvent~~ at the time of sample preparation from the same solvent source used in sample preparation.

7.3.2. Evaluate the negative QCC to ensure that the instrument and solvent are free of any controlled substance, any substance being identified in the sample and any substance that may interfere with the identification of sample component(s).

7.3.2.1. Note the presence of large amounts of common gas chromatography peaks (e.g., siloxanes) in the GC-MS activity log and notify the Drug Chemistry Technical Leader.

7.3.2.2. Record all negative QCC's results (pass/fail) and any comments on the GC-MS activity log

7.4. Evaluate and prepare samples prior to injection to avoid overloading, extreme pH, oils, sugars and compounds known to be retained in the instrument.

7.4.1. At a minimum, filter solid samples with solvent to prevent particulate matter and undesired compounds from being introduced into the instrument (e.g., sugars). Particulate matter should not be visible in an autosampler vial.

7.4.2. Refer to the Drug Chemistry Unit Technical Procedure for Extractions for additional sample preparation.

7.4.3. Extract/convert sulfates prior to introduction into the instrument.

7.4.4. If a derivatizing agent is used the controlled substance reference material must be derivatized contemporaneously.

7.4.4.1. The mass spectrum of the derivatized reference material must be compared to published spectral data from an informed treatise generally accepted in the field and found to be substantially comparable, i.e., equivalent.

7.5. Use the current date in the names of sequences. Sequences need not be archived. Upon completion of each sequence, print the sequence log and store in the GC-MS logbook.

7.6. Include the entire CCBI assigned case file number in the data file name and any additional information needed to uniquely identify the sample.

7.6.1. Data files associated with casework and performance checks shall not be deleted or overwritten.

7.6.2. The Drug Chemistry Technical Leader shall archive data files annually and label with the instrument serial number and dates. Store archived files near the instrument.

7.6.3. Notify the Drug Chemistry Technical Leader if the data storage location becomes full.

7.7. The GC-MS provides retention time data and mass spectral data. Refer to the Drug Chemistry Unit Technical Procedure for Drug Chemistry Analysis. Evaluate the chromatogram and spectra for each peak.

7.8. Record in the case record the:

7.8.1. Total Ion Chromatogram (TIC) for the corresponding blank.
7.8.2. Sample TIC.
7.8.3. Mass spectra of significant peaks.
7.8.4. Expanded mass spectra of any phenethylamines.

7.9. Mass Spectral Comparison

7.9.1. For a positive mass spectral comparison, the sample mass spectrum must be substantially comparable, i.e., equivalent, to that of primary or secondary reference material.

7.9.1.1. Record in the case record the mass spectrum of the reference material with the supplier and lot number or other Drug Chemistry Unit designation. Library search results may be included.

7.9.2. If a derivatizing agent is used the mass spectrum of the sample mass spectrum must be compared must be compared to contemporaneously prepared derivatized reference material and found to be substantially comparable, i.e., equivalent.

7.9.2.1. Record in the case record the mass spectrum of the reference material with the supplier and lot number or other Drug Chemistry Unit designation and the supplier and lot number of the derivatizing agent.

7.10. GC Retention Time (RT) Comparison

7.10.1. For a positive GC RT comparison of compounds with a retention time of 10 minutes or less, the difference between the sample retention time and a primary or secondary reference material retention time must be 0.10 minute or less. For a positive GC RT comparison of

compounds with a retention time greater than 10 minutes, the percent difference between the sample retention time and a primary or secondary reference material retention time must be 1.0% or less.

7.10.1.1. The chromatographic peaks must be visually smooth and symmetrical.

7.10.1.2. The reference material must be run within thirty days before or after the case sample.

7.10.1.2.1. If the reference material is a component of the monthly QCC solution, the retention time may be used for the month in which it was run plus the first seven calendar days of the following month.

7.10.1.2.2. There must not be any column maintenance performed between the analysis of the sample and reference material.

7.10.1.3. Record in the case record:

7.10.1.3.1. Reference material TIC with the retention time(s) displayed.
7.10.1.3.2. Reference material mass spectrum and any other significant peaks with the retention time(s) displayed.
7.10.1.3.3. Reference material standard supplier and lot number or other Drug Chemistry Unit designation.

7.10.1.3.4. The percent difference of the reference material and sample retention times, rounded to one decimal place, refer to 11.7.

8. Calculations

8.1. Percent Difference Calculation, round to one decimal place, refer to 11.7.:

|(reference material RT – sample RT)| / (reference material RT) * 100

9. Limitations

9.1. The GC-MS methods described in this procedure shall not be used to distinguish between optical isomers.

9.2. Introduction of improperly prepared samples may lead to poor sensitivity and carryover.

10. Safety

10.1. Refer to the CCBI Crime Laboratory Safety Manual.

10.2. Handle syringes with care to avoid punctures.

10.3. Use extreme caution handling/installing/removing/transporting compressed gas cylinders. Cylinders shall not be moved without the cylinder cap securely in place.

10.4. Gas Chromatograph and Mass Spectrometer may be extremely hot. Avoid touching hot areas and wear protective gloves while performing maintenance.

11. References

11.1. Agilent 6890 GC Instrument Manuals.

11.2. Agilent 5975 MSD Instrument Manuals.

11.3. Moffat, A. C., et al., eds. *Clarke's Isolation and Identification of Drugs.* 2nd Edition. London: Pharmaceutical Press, 1986.

11.4. Moffat, A.C., et al., eds. *Clarke's Analysis of Drugs and Poisons.* 4rd Edition. London: Pharmaceutical Press, 2011.

11.5. Skoog, Douglas A., F. James Holler and Timothy A. Nieman. *Principles of Instrumental Analysis*. 5th Edition. Garcourt Brace & Company, 1998.

11.6. *Agilent GC-MSD ChemStation and Instrument Operation Student Manual Course Number H4043A Volume 1*, Revision E.02.xx. Agilent Technologies: printed February 2008.

11.7. *Guide for the Use of the International System of Units (SI).* NIST Special Publication 811, 2008 Ed., (March 2008; 2nd printing November 2008). p.43.

12. Records

12.1. GC-MS Weekly Maintenance Log
12.2. GC-MS Monthly Maintenance Log
12.3. GC-MS BiannualMaintenance Log
12.4. GC-MS Annual Maintenance Log
12.5. GC-MS Daily QCC Log
12.6. GC-MS Monthly QCC Log
12.7. GC-MS Activity Log
12.8. Case record

Revision History		
Effective Date	Version Number	Reason
1/1/13	1	ISO Compliance
1/8/13	2	Correction to 7.10.1-requirements for GC retention time comparison.
8/7/13	3	Incorporation of Uncertainty of Measurement and Measurement Assurance
2/16/15	4	Updated lines 5.1, 5.7.8.2, 6.4.2, 6.4.4.1, 6.4.6, 6.4.7.1, 7.3, 7.3.1 and 7.5.