Controlled Substance Analysis Standard Operating Procedures
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General Evidence Procedures
This section is intended to provide guidance when accepting, storing, and returning evidence.

1. Evidence Acceptance

1.1.0 A completed Lab Request Form, District Attorney's office request, or PLIMS electronic service request must be received before analysis can proceed on a case. Incomplete Lab Request Forms or service requests will be rejected.

1.1.1 All evidence submitted to the lab will follow PM 4.2, Acceptance of Evidence. Additionally, basic forensic evidence handling expands these requirements to ensure the proper labeling of test items, prevention of contamination, and the securing of evidence and documentation.

1.1.2 Sharp objects such as syringes are <u>not</u> routinely accepted for analysis. They should be properly secure using a Sharps container or other appropriate method

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

1.1.4 Wet items of a large quantity will not be accepted by the Chemistry Section, as there is no suitable place to dry the material prior to analysis.

1.1.5 In a case with multiple items, those of schedule I, II, or III may be analyzed before other items in a case.

1.1.6 In cases where the item will not exceed a statutory threshold, the item will be analyzed for simple possessions only see *sampling (3.1.9)*.

1.1.7 Inappropriately packaged evidence should be returned to property control, see 1.2.14 of this section or PM 4.7.

1.2 Evidence Handling

1.2.1 The chain of custody is recoded electronically in the PLIMS system by scanning the unique barcode label attached to the evidence.

1.2.2 The person receiving the evidence is responsible for ensuring that the complaint number in the PLIMS system matches the evidence labels affixed to the item and that the evidence is sealed and properly stored.

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

1.2.4 Repackaged evidence will be marked using the analyst's initials and by placing a new barcode evidence label on the exterior packaging. Packaging should be resealed according to PM 4.4.4.

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

1.2.6 Special care will be taken to prevent cross contamination. These precautions include opening only one evidence item at a time and cleaning the test surface regularly.

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

1.2.8 Outer packaging/containers will be closed and secured through the use of tape with or without staples or heat sealed.

1.2.9 Outer packaging seals will contain the initials and code number of the person sealing the package, along with the date of the seal.

1.2.10 A copy of the Property Report or PLIMS electronic chain of custody record showing when the evidence is returned to Property Control, will be kept as part of the case file.

1.2.11 Per the analyst's discretion, when any significant weight, count, item descriptions, or extra item discrepancies are found the officer and Chemistry supervisor should be notified.

1.2.12 For minor discrepancies, a notation on the worksheet should suffice.

1.2.13 Unacceptable evidence which cannot be immediately corrected will be returned to Property Control with an Evidence Rejection Form (follow PM 4.7). A copy of this form will be kept in the case file (when available) or the chemistry section. Submissions returned to contributor without testing should have appropriate documentation in the file.

*Note: All notes, worksheets, photos, printouts, and any other hardcopy documentation will be scanned and attached to the assignment in the PLIMS system.

References

Master, Nancy E. 1999. What is Aspergillus? (Part1) International Association for Property and Evidence--*Evidence Log*. No. 3.
Master, Nancy E. 1999. What Can Be Done About Aspergillus? (Part 2) International Association for Property and Evidence--*Evidence Log*. No. 4.

2. Analysis

Identifications will be made using techniques accepted in the field of forensic science. The scope of the testing will be sufficient such that in the absence of the analyst, another competent analyst could evaluate what was done and interpret the data.

2.1 Analytical Standards

2.1.1 Flexibility and innovation are desirable qualities in relation to analysis design/ instrument parameters while remaining within scientifically accepted norms for recognizing compound identity.

2.1.2 The examination shall be designed so as to find the presence of normally encountered controlled substances when present at 'street level' concentrations in a technically homogenous sample.

2.1.3 Exhaustive testing should be balanced with the necessity for productivity. As a general guide, the minimum amount of items and testing should be performed to meet statutory guidelines while leaving sufficient sample for additional testing or required by subsequent information or requests.

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

2.1.5 It is the intention of the Chemistry Section to follow the minimum standards of drug identification as established by SWGDRUG.

2.1.6 A minimum of two separate tests with positive results are required to report a substance, controlled or non-controlled.

2.1.7 Preliminary color testing should be done when possible in addition to two separate tests with positive results. At least one of the two tests must come from Category A but the second test could come from Categories A or B using the table below as a guide:

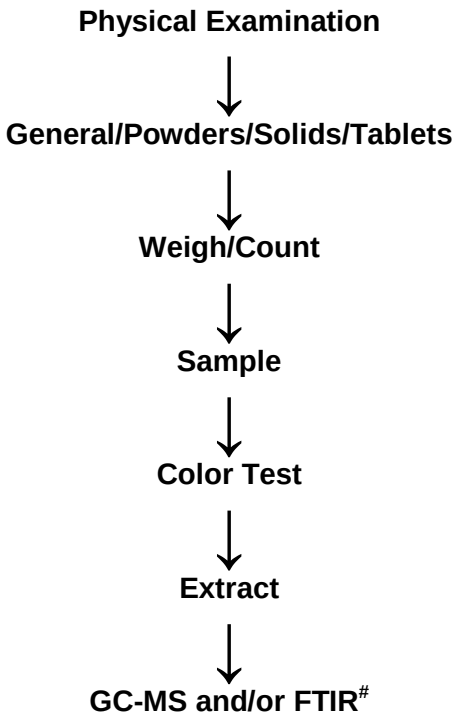
Category A	**Category B**	**Category C**
Mass Spectrometry	Gas Chromatography	Color Tests
Infrared Spectrometry	Pharmaceutical Identifiers	UV Light
	Macroscopic/Microscopic Identifiers	

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

2.2 Analytical Schemes

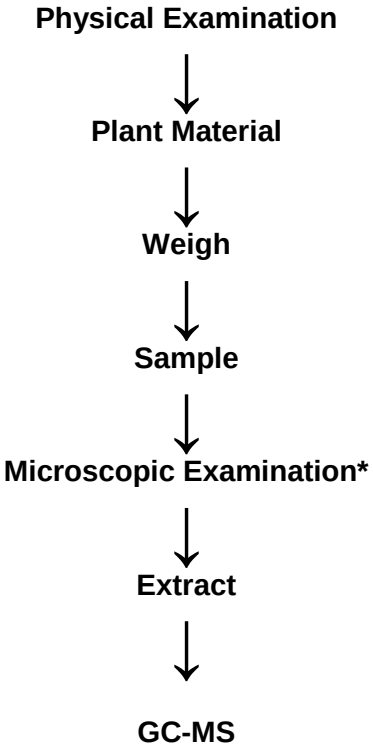
The following flow charts are the analytical schemes to be used for controlled substances. These flow charts are to be used as a general guide and deviations are up to the discretion of the analyst. For these cases the flowchart for general unknowns can be followed and any modifications noted in the case file. It should be noted that sample size or other circumstances may require rearrangement or modification of one or more steps.

2.2.1 General/Powders/Solids/Tablets



required for Federal cases to determine Cocaine HCl or Cocaine Base

2.2.2 Marijuana



*
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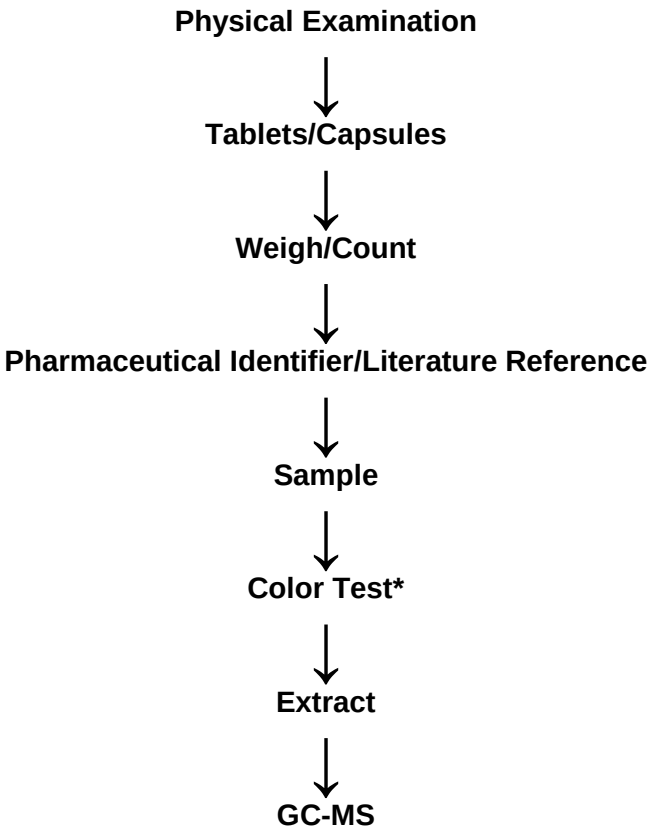
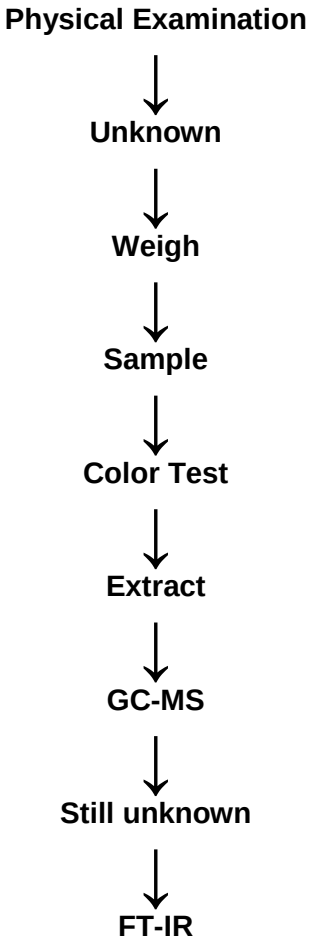
*
*
* If appropriate

2.2.3 Pharmaceutical Preparations



* If appropriate

2.2.4 Unknowns



2.3 Preliminary Testing

Color Testing

Color tests are intended to be a screening agent at the beginning of an analysis. It is acceptable to perform them either in a clean porcelain or disposable plastic spot plate or alternatively in a disposable culture tube.

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

2.3.2 It is suggested that the reagent be added to a clean spot plate well first to demonstrate no contamination of the well, but this may not always be practical depending on the sample type. The order of placing the reagent and the sample in the spot plate well will be left up to the analyst's discretion.

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

References

Moffat, A. C., Jackson, J. V., Moss, M. S., and Widdop, B. *Clarke's Isolation and Identification of Drugs*. The Pharmaceutical Press, London, 1986.
Sigma Chemical Company 1993. Forensic Chemistry Drug Stat Kit-Cobalt Thiocyanate Test Reagents. Sigma Technical Bulletin #QTCOCA.
Sigma Chemical Company 1993. Forensic Chemistry Drug Stat Kit-Duquenois Test Reagents. Sigma Technical Bulletin #QTMJ.
Sigma Chemical Company 1993. Forensic Chemistry Drug Stat Kit-Marquis Test Reagents. Sigma Technical Bulletin #QTOPI.
Sigma Chemical Company 1993. Forensic Chemistry Drug Stat Kit-Simon's Test Reagents. Sigma Technical Bulletin #QTMETH.
O'Neal, Carol L., Crouch, Dennis J., and Fatah, Alim A. 2000. Validation of twelve chemical spot tests for the detection of drugs of abuse. Forensic Science International 109:189-201.
Koppenhaver, David. 1997. GHB Color Test. Microgram Journal XXX: 130.
Assorted Microgram Journals.
Assorted Journals of Forensic Science.
Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG) Recommendations, 4th edition, 2008.

Microscopic Analysis

Microscopic examination is used for plant material or suspected marijuana.

2.3.4 Cystolithic hairs are unicellular, 'bear claw' shaped hairs with a cystolith at the base. They are found in greatest abundance on the upper side of the leaf. Simple or covering hairs are found on the underside of the leaf. Glandular hairs have a short stalk and a bulbous end that secretes resin which contains THC.

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

2.3.6 At least two of the above features are needed for a preliminary positive.

References

Sigma Chemical Company 1993. Forensic Chemistry Drug Stat Kit-- Duquenois Test Reagents for Marihuana. Sigma Technical Bulletin #QTMJ.
Smith, R.N. 1974. A Brief Note on the Response of Some Essential Oils and Extracts of Vegetable Origin to the Duquenois-Levine Test for Cannabis. Journal of the Forensic Science Society 14: 191-194.
Kovar, K. et al. July 1989. Concerning the Duquenois Test for Identification of Hashish and Marihuana. Microgram Journal XXII: 129.
Yinon, Jehuda. Forensic Mass Spectrometry. CRC Press, Florida.
Microgram Bulletin article March 2009.
Journal of Mass Spectrometry (2009).

UV Light

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

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

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

References

Phillips, William, et al 1974. Distinction of Lysergic Acid Diethylamide and Lysergic Acid
Moffat, A.C. Clarkes Isolation and Identification of Drugs. 2nd Ed. London: The Pharmaceutical Press, 1986

2.4 Confirmatory Testing

2.4.1 Gas Chromatography/Mass Spectrometry

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

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

2.4.1.2 Confirmation of an unknown spectrum is done by direct comparison with a reference standard, library spectrum, or published reference spectrum.

2.4.1.3 Samples will be dissolved in a suitable solvent (*methanol/chloroform/ hexane*).

Samples will be run using the appropriate method based on the type of sample. See Appendix F for a list of methods and which samples should be run using which method.

2.4.1.4 Typically, controlled substance solutions contain 1 mg/mL of standard material. Variations in standard concentration are based on instrumental conditions, identity of material being analyzed and other factors.

Standards are verified by GC-MS before use.

References

Agilent website.
Instrument owner's manual/software.

2.4.2 Infrared Spectroscopy

The purpose of this document is to outline the general method of analysis for examination of controlled substances and drug evidence by Infrared Spectrophotometry.

This method of spectral analysis is based on the molecular vibrational energies of an organic compound. As the different wavelengths of infrared light pass through a molecule they are absorbed by the molecule or transmitted by the molecule according to the different vibrational frequencies within the molecule. These absorbed or transmitted frequencies can then be recorded electronically and a resulting spectrum generated.

The mid-infrared region extends from 4000 to 400 cm^{-1}.

A Fourier transform infrared (FTIR) spectrometer is a multiplex instrument, one in which all elements of the signal are observed simultaneously. Fourier transform is a signal decoding process used for data processing.

The FTIR spectrometer will be used with the Attenuated Total Reflectance (ATR) accessory.

A FTIR spectrum will be obtained whenever a substance is suspected to be cocaine for Federal cases to determine between Cocaine HCl and Cocaine Base.

FTIR spectrum will be collected when an item is a complete unknown, ie has not been identified by GC/MS analysis.

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

Comparison of the analytical data to approved reference standards/publications will be the means of identification.

IR standards may be used neat.

Procedure

2.4.2.1 Obtain a background spectrum on the clean accessory. If the accessory exhibits uncharacteristic absorbance, clean the accessory and repeat.

2.4.2.2 Before each sample, collect a ‘blank’ spectrum on a clean accessory.

2.4.2.3 Place sample on the diamond surface and apply pressure for solids. No pressure is necessary for liquid samples. Evaporate any unwanted solvents.

2.4.2.4 Obtain spectrum that includes the region between 4000 and 600 cm^{-1}, with a minimum resolution of 4 cm^{-1}.

2.4.2.5 After analysis is complete, clean the diamond surface and pressure device if applicable with isopropyl alcohol or other appropriate solvent.

3. Measurement Practices

3.1 Sampling

3.1.1 Sampling is a process whereby a portion of an evidence item is taken for testing to serve as a representative of the whole. To insure that analyses are thorough and to answer the requirements of North Carolina law, sampling in multiple item cases/mixtures should be regulated and defined.

3.1.2 It is up to the analyst's discretion as whether to adopt a statistical or non-statistical sampling plan.

3.1.3 Each sampling plan represents the minimum to be sampled. An analyst may always choose to sample more items.

General Sampling Plan/Non-statistical Sampling Plan

- Distinctive items shall be separated for analysis/sampling.
- Distinct packages with obvious cross contamination may be combined at the discretion of the analyst.
- Whenever possible and practical, the analyst shall leave a portion of the original sample for potential re-analysis and court viewing. This may not be possible for items containing a trace amount.

3.1.4 Single Item Case – the specimen will be fully analyzed.

3.1.5 Multiple Item Cases

Within a distinct package, like items may be combined, but visually different items shall be separated by common characteristics when possible (size, color, shape, etc) to obtain a homogenous population.

Clandestine Preparations:

Chunky material and powders in proximal contact (co-mingled) or with similar physical characteristics (color, consistency) may be considered as components of a mixture and may be treated as a singular item/sub-item. Co-mingled clandestine tablets that bear consistent physical characteristics including color, shape, imprint, and consistency, may be combined into singular items/sub-items.

Multi-packet Sampling Policies:

a. Any chunky material with significantly dissimilar physical characteristics even though they are in proximal contact shall be treated as separate items/sub-items.

b. Chunky material, powders, and liquids that do not bear enough unique characteristics to be combined based on their physical characteristics alone; therefore, distinct packages of chunky material, powders, or liquids within one evidence container and with similar physical characteristics may not be combined.
c. Clandestine or unlabeled capsules/pills shall be considered individual distinct packages and may be combined.
d. Items that contain 2 or more distinct packages that contain visually similar samples and exceed a threshold value may be processed by complete analysis of enough packets or sub-items to exceed the threshold weight plus the total uncertainty.
e. Items that contain distinct packages that contain visually similar samples and are below a threshold value may be processed:
    i. by complete analysis of each packet or sub-item, or;
    ii. testing enough packet/sub-items to ensure that the needs of the investigation/case are met.

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

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

3.1.8 If the uncertainty of measurement lowers the total net weight to below a statutory threshold, then further analysis must be performed to meet the threshold randomly selected packets/sub-items. All others may be reported in totality as No Analysis Performed.

3.1.9 When the total units in an item do not exceed a statutory threshold, a complete analytical format of tests may be performed on a minimum of 5 (if 5 or more are present). The examiner has the discretion of choosing which items/exhibits to test.

Pharmaceuticals

3.1.10 Due to the unique physical identifiers present in pharmaceutical preparations, consistent sample population can easily be determined.

a. Combine all tablets with identical physical characteristics and/or pharmaceutical identifiers into one item or sub-item. Do not combine distinctly separated or packaged sub-items.
b. When applicable, use the pharmaceutical identifiers to determine the probable identity of any controlled substance present.
c. Determine the total weight of the submission and net weight of one pill.
d. Randomly select (sample) at least one pill for additional testing to include structural determining testing.
e. Report the net weight of the item and the count and/or net weight of the sample selected for full testing in the case notes and on the laboratory report. Pharmaceutical preparations in which the imprint, markings, and

physical characteristics cannot be identified using an approved reference shall be analyzed as if they were clandestine preparations.

Bulk Materials

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

Hypergeometric Sampling Method

3.1.12 Hypergeometric sampling is a statistically based model involving a defined confidence level with an associated probability of finding failures in a population.

If preliminary testing indicates that no controlled substances are detected, the hypergeometric sampling plan may be adopted.

The appropriate number of specimens within the population will be randomly selected to give a 95.45% confidence level that at least 90% of the population contains the analyte of question.

Record the number of specimens indicated by the table in Appendix D on the worksheet.

Each specimen sampled, as determined from the table, will be analyzed separately and fully.

3.2 Weighing Practices

It may be necessary to analyze additional items to satisfy weight requirements mandated by law. All solid and vegetable material tested shall be reported as a net weight. A net weight is the weight of the substance without any packaging. The gross weight is a weight with packaging or miscellaneous materials. The net weight will be determined in the most accurate manner possible considering the packaging of the evidence items.

3.2.1 Wet evidence should be dried prior to being weighed.

3.2.2 An attempt will be made to minimize sample loss at the same time minimal effort will be put forth in the removal of packaging from sample.

3.2.3 Weights shall be reported in the units and significant digits recorded from the balance.

3.2.4 The drug worksheet shall reflect the actual reading(s) from the balance.

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

3.2.6 A net weight should be reported in addition to the tablet count for illicit and pharmaceutical tablets/capsules. The net weight of illicit capsules shall include the weight of capsules and contents. The reported weight of illicit tablets/capsules will reflect only those that are visually consistent with the tablets/capsules tested.

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

3.2.8 Solid material present in an amount less than ± 0.03 grams may be reported as “trace amount” or “residue”.

3.2.9 Cases involving quantities that exceed the capacity of the personal balances should be weighed on other provided balances following the use protocols (verification check at the time of weighing).

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

Multiple Samples

3.2.11 If there are different types of samples to be analyzed, at least one of each type must be weighed.

Trafficking Limits

3.2.12 In instances where statutory requirements or state sentencing guidelines designate weight thresholds, sufficient specimens will be weighed and analyzed to exceed the threshold, see Appendix E. The method used to generate the reported value must have an uncertainty that can be defined, and (if sufficient sample exists) the data reported can have no reasonable possibility of being below the legal limits.

3.2.13 As a guide, case materials which show a calculated sample weight within the uncertainty range of a legal limit as determined by representative sampling must be weighed to fulfill the weight threshold. Further weighing must be conducted to establish proximity to legal limits (i.e. a sufficient number of separate packages or all packages must be opened to demonstrate a sample weight above the legal limit).

Example:

If the statutory limit is 4 grams for trafficking and the total gross weight of 100 samples is 7.60 grams
10 samples out of 100 are weighed and color tested and are consistent
10/100 = 0.41 grams net weight
Which equals an average weight of 0.041 grams per sample,
multiplied by 100 equals a total calculated net weight of 4.10 grams
The calculated net weight is less than the statutory weight limit plus five percent (4.20 grams).

3.2.15 The same concept will apply to counting of items (weight count) or measuring of items (LSD paper) when legal quantity limits are involved.

3.2.16 If analysis reveals a mixture of trafficking drugs with different penalties (such as MDMA mixed with methamphetamine), the analyst will prove both drugs, the one with the higher schedule, or contact the prosecuting agency to determine if identification of both drugs is required.

3.2.17 If the weight or number of illicit tablets containing a trafficking drug exceeds statutory limits, at least one tablet of each type/color/logo needed towards proving the statutory weight will be tested.

3.3 Uncertainty of Measurement (UOM)

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

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

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

3.3.2 Uncertainty budgets for individual balances should be re-evaluated on an annual basis. The actual uncertainty budget table calculations/data for each balance will be maintained in separate binder located in the Chemistry Section of the laboratory.

3.3.3 Calculations used to estimate the uncertainty should be rounded up to be conservative. The units of uncertainty values should be measured (converted) in the same units.

3.3.4 The final UOM shall not exceed two significant figures.

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

Definitions for Uncertainty of Measurement

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

Definitions

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

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

The mean is defined as the sum of the measured values divided by the total number of values: $X = \frac{\sum x_i}{n}$

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

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

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

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

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

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

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

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

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

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

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

Estimating the Uncertainty of Measurement

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

Example of Budget table

Component	Value	Units	Distribution	Type	Divisor	Degrees of Freedom (n-1)	Standard Uncertainty	Relative Contribution
Process Reproducibility	0.018	g	normal	A	1	12	0.018	44.7 %
Linearity	0.02	g	rectangular	B	√3	Infinite	0.012	29.8 %
Readability at Zero	0.01/2	g	rectangular	B	√3	Infinite	0.003	7.4 %
Readability at load	0.01/2	g	rectangular	B	√3	Infinite	0.003	7.4 %
Balance Calibration	0.0086	g	normal	B	2	infinite	0.0043	10.7%

uncertainty								
	Combined Standard Uncertainty (Uc)						0.0403	

Calculation of Combined Uncertainty

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

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

For a Single Item, the combined uncertainty is the same as uncertainty for the measurement.

For Multiple Items
When multiple weights are totaled, the uncertainty associated with each individual weight must be accounted for in the total uncertainty calculation.

$$U_{combined\ total} = \overline{n} * U_{combined}$$

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

Calculation of Expanded Uncertainty

The expanded uncertainty is used as a measure of uncertainty that defines a confidence interval about the measurement result. It is the combined uncertainty multiplied by the confidence factor (k).

$U_{expanded} = U_{combined}$ (k) k = 2, at the 95.45% confidence level
For the single item example, the expanded uncertainty at the 95.45% confidence level is:

$$U_{expanded} = 0.04g\ (2) = 0.08g$$

For a multiple items example where the number of items is 5, the expanded uncertainty at the 95.45% confidence level is:

$$U_{expanded} = n * U_{combined} *(k)$$

$$U_{expanded} = 5* 0.04g\ (2) = 0.4g$$

Recording and Reporting Weights with Uncertainty

The measurement of uncertainty will be recorded in the case notes and on the report as the total weight ± the expanded uncertainty at the 95.45% confidence level. A statement will be added to the report, 'All ± weights are at a 95.45% confidence level.'

References

American Society of Crime Laboratory Directors/Laboratory Accreditation Board (ASCLD/LAB) manual.

ASCLD/LAB-*International* Guidance on Measurement Traceability, May 2013.

ASCLD/LAB-*International* Guidance on Measurement Traceability-Measurement Assurance, May 2013.

ASCLD/LAB-*International* Guidance on the Estimation of Measurement Uncertainty – Overview, May 2013.

ASCLD/LAB-*International* Guidance on the Estimation of Measurement Uncertainty –Annex B Drug Chemistry, May 2013.

Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG) Supplement Document SD-3, Examples of Measurement Uncertainty for Weight Determinations, 7-7-2011.

ASCLD/LAB Measurement Confidence 100 and 200 level courses, 2013.

ISO/IEC 17025: 2005

4. Reporting and Documentation Procedures

4.1 Case Files / Electronic Case Records

4.1.1 The case file or electronic case record will contain all documentation related to a request for analysis and all communication about that case.

4.1.2 At a minimum, the case file/electronic case record will contain the analyst's notes, charts, graphs, data, worksheets, the report, and a copy of the property report showing evidence transfer as chain of custody.

4.1.3 Each page within the case file/electronic case record should have at a minimum the lab number for that case and the analyst's initials, except for administrative documents that are stapled together and then the lab number may be on the first page only and the analyst's initials must be on all pages.

4.1.4 All significant and meaningful communication about the case (verbal or written) must be documented in writing with date, analyst initials, and lab number.

4.1.5 Completed physical case files will be stored in the file room by complaint number or administratively approved archive storage. Electronic case records will be stored in the PLIMS system.

4.2 Case Notes, Worksheets, & Photographs

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

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

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

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

4.2.5 All handwritten records will be generated in ink.

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

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

4.3 Reports

4.3.1 At minimum, the identity of the item with the highest schedule will be reported whenever confirmed.

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

Examples include:

● Item(s) ___was/ were analyzed by color test(s) and Gas Chromatography-Mass Spectrometry (GC-MS).

● Item(s) ___ was/were analyzed by color test(s), Gas Chromatography-Mass Spectrometry (GC-MS), and Infrared Spectroscopy (FTIR).

● Item(s) ___ was/were identified by their pharmaceutical imprint and Gas Chromatography-Mass Spectrometry (GC-MS).

● Item(s) ___ was/were analyzed by microscopy and Gas Chromatography-Mass Spectrometry (GC-MS).

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

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

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

4.3.5 The term *residue* or *trace* may be used instead of a weight for values <0.03 grams.

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

4.3.7 Liquid samples of suspected LSD will be reported in milliliters. One drop will be considered to be approximately 0.05 mL unless the dropper “device” is tested.

4.3.8 Containers with an intact manufacturer’s seal can be assumed to contain the stated volume.

4.3.9 Pharmaceutically prepared tablets/capsules should be reported to include the count and weight. Illicit tablets that meet a statutory tablet count should also be reported to include the count and weight.

4.3.10 Items received but not analyzed will be listed on the report.

4.3.11 The following are possible options for reporting results:

- "*confirmed substance*"
- No controlled substance detected
- Inconclusive *(reason on work sheet)*
- Quantity not sufficient for positive identification
- Item not suitable for analysis
- No analysis

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

5. Quality Assurance

Instruments located within the Chemistry section will only be operated by authorized and trained personnel.

5.1 Each instrument will have a QC notebook and maintenance log.

5.1.1 Any instrument that fails the quality control checks or gives suspect results will be placed 'out of service' until the necessary repairs or maintenance are made to correct the problem.

5.1.2 Any repairs will be recorded in the instruments maintenance log.

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

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

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

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

5.2 Reagents

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

5.2.2 When a reagent/solution is made, the lot numbers should be recorded on the Lab Solution Form and the reagent should be assigned a new lot number consisting of the date it was made and tested as well as the analyst's initials (example 062811AC).

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

5.3 Standards

The Chemistry Section keeps both primary and secondary reference standards.

5.3.1 A primary standard is one obtained from a reputable manufacturer.

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

5.3.3 All primary standards must be authenticated at time of use. Anytime a standard is authenticated a Standard Authentication Form must be completed and placed in the Standard Authentication Notebook after being reviewed.

5.3.4 A certificate of analysis received with a standard will serve to establish the quality of the standard.

5.3.5 Upon analyzing the standard the analyst should also check to ensure that data acquired is consistent with the literature for that standard and the analyst's expectations.

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

- Manufacturer's certificate of analysis
- At least one set of category A instrumental printouts

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

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

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

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

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

- Category A instrument print outs

- Additional printouts from a category A or B analysis (or C, see not above)
- Literature reference to support authenticity

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

5.3.13 Standard Authentication packets will be kept in the laboratory for at least five years and archived as needed.

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

Expiration Dates

All standard containers should be labeled with an expiration date. (PM5.2)

1. Standards (solids)…… expire 5 years from date opened*****
2. Standards (liquids)…… expire 2 years from date opened*****
3. Positive Controls (e.g. Marihuana & Heroin/Cocaine mix) expire 1 year from date prepared
4. Negative Controls (i.e. Oregano & Inositol)……. expire 5 year from date opened
5. 1 mg/mL solutions. …… expire 1 year from date prepared*****

*******Use the manufacturer's expiration date if provided*

Re-validation

Standards should be re-validated with the same method in which they were initially validated. All chemicals should be re-validated only one time.

1. Standards (solids)…….. Extended for 2 years
2. Standards (liquids)…….. Extended for 1 year
3. Positive controls (e.g. Marihuana)…… Extended for 6 months
4. Negative Controls (e.g. Oregano & Inositol)…..…… Extended for 6 months
5. 1 mg/mL solutions…….. Extended for 6 months

5.4 Blanks

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

5.4.1 Running GC/MS Blanks

5.4.1.1 A blank consisting of the solvent used to dissolve the samples should be run before every case sample.

5.4.1.2 The injection order when running samples with standards/positive controls should be either 'standard, blank, sample' or 'blank, sample, standard'.

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

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

5.4.1.5 A blank should be re-run if a peak in the blank is close to a peak of interest, ±0.05 minutes.

5.4.2 Running FTIR Blanks

5.4.2.1 A blank spectrum of the clean accessory will be collected before every case sample. If the first blank spectrum does not fall within acceptable range (5.9.8), another blank spectrum will be collected after cleaning the accessory.

5.5 Color Tests

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

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

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

5.5.3 Reagents should be made in quantities to minimize waste. The shelf-life of a reagent may vary. The reagent is considered to be viable as long as it reacts appropriately in the QC check. Reagents that fail to react will be discarded appropriately.

5.5.4 When a reagent is made fresh, the lot numbers will be recorded and the reagent should be assigned a number consisting of the date it was made and tested as well as the analyst's initials (example 041311JL).

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

A positive and negative control will be run using the table below as a guide.

	Check Compound		
Reagent	Positive Control	Positive Result	Negative Control
Cobalt Thiocyanate	Cocaine	Blue	Inositol
Marquis	Meth	Orange	Inositol
Other color reagents	See reference literature for appropriate choice		

References

Moffat, A. C., Jackson, J. V., Moss, M. S., and Widdop, B. *Clarke's Isolation and Identification of Drugs*. The Pharmaceutical Press, London, 1986.

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

5.6 Equipment Maintenance

Any equipment used for measuring that fails a performance check or calibration must be taken immediately off-line and reported to section supervisor or designee.

External Calibrations shall be performed by an ISO 17025 accredited company when available.

- When equipment is checked or calibrated by an external organization, the procedures used will be determined by the external organization.
- Documentation of the check or calibration shall be provided

5.6.1 Weights

5.6.1.1 Transportation and Handling of Weights

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

5.6.1.2 Definitions:

Primary mass standards – Certified NIST traceable weights used to verify secondary mass standards. Primary mass standards will meet or exceed ANSI/ASTM E617 Class 1 standard (ex. UltraClass).

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

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

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

ANSI/ASTM E617 Class 1 tolerances

Nominal value	Tolerance
1 gram	0.034 mg
10 grams	0.050 mg
100 grams	0.25 mg

5.6.1.4 If at any time a primary mass standard is dropped, becomes damaged or potentially altered in any manner or does not meet acceptable tolerance level, it must be immediately taken out of service. The affected mass standard must be recertified by an external agency prior to being placed back into service or replaced.
All weights used to verify balance calibration (secondary mass standards) shall be evaluated annually to determine if the weight meets acceptable criteria - *see QM 5.6.3.1.*

5.6.1.5 Process for checking Secondary weights:

For each secondary standard being checked, a primary mass standard is weighed. That weight is recorded. The balance must provide a reading that is within the acceptable tolerance listed for that weight. Once the balance has been determined to be sufficiently accurate using the primary mass standard, the secondary calibration weight is weighed, and that weight is recorded. If the measured weight of the secondary calibration standard falls within the declared tolerances of the primary calibration standard (i.e., within ± 1%), the standard is suitable for use in performing the routine calibration of balances. If the measured weight does not meet the criteria, it must be replaced or adjusted and recertified by an external agency. If at any time a secondary calibration weight is dropped or potentially altered in any manner, it must be rechecked using this procedure.

5.6.2 **Verification of Balances**

Each analyst or employee is responsible for performance checking any balances he/she used during the course of casework, or in the preparation of standards, controls, calibrators and reagents to be used during the course of casework, on the day(s) the balance is used.
Additional calibration verification will be performed if the balance has been moved or any time the operability of the balance is called into question.

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

- Personal balances will be checked weekly using a set of secondary mass standards (minimum ASTM class 2).
- A monthly performance check will be done using surrogate weights (plant material and powder).
- Each personal balance will be checked quarterly using the primary mass standards (Ultraclass weights).

- Each personal balance will be calibrated annually by an approved outside vendor. The annual calibration may be used in place of the scheduled in-house check for that time period.

5.6.2.2 The actual readings of all performance checks will be recorded in the analyst's point of use (POU) log or the balance log.
Weight measurements should be within current acceptable ranges (*see Balance Performance Verification Log or POU for acceptable ranges of each balance)*. If a weight measurement exceeds the acceptable range, confirm that the balance is level and/or perform the internal balance re-calibration then repeat all weight measurements again.

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

5.6.3 Microscope and alternate light source (ALS) repairs are made as needed. Microscope is serviced once a year by an ISO 17025 accredited company.

5.6.4 Oven, Refrigerator, and Freezer.
The temperature of the oven, refrigerator, and freezer are monitored and recorded.

- For refrigerators, the temperature shall be between 2 – 8 º C.
- For freezers, the temperature shall be below -5 º C.

Once a year each thermometer is checked against a NIST certified thermometer

5.6.5 Adjustable volume and single volume pipettes should be cleaned by wiping the surface with Ethanol or 10% chlorine bleach solution. Allow to air dry. Clean monthly or as needed.
Precision liquid processor (i.e. Hamilton Microlab Diluter) should have surface wiped with Ethanol or 10% chlorine bleach solution and deionized water. Allow to air dry. Purge, clean, and decontaminate all portions of the tubing and syringes. Use a cleaner that is compatible with the fluids previously run through the system. Tubing that is in poor condition should be replaced. Clean monthly or as needed.

Precision liquid processor should be calibrated and/or performance verified once a year by a certified vendor. In-house performance verification should be done every year.

5.7 Validation

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

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

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

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

5.8 Gas Chromatograph/Mass Spectrometer (GC/MS)

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

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

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

5.8.4 Evaluating Chromatography Performance

5.8.4.1 Chromatographic performance will be evaluated weekly by tuning the GC/MS and running caffeine. Mixed standards as positive controls will be run monthly using the drug scan method (DRGSCN) or equivalent.

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

5.8.4.3 The GC/MS test mix can contain any of the following but not limited: methadone, caffeine, cocaine, codeine, delta-9-tetrahydrocannabinol, 6-monoacetylmorphine, heroin, etc. The positive control is considered acceptable if all expected components elute with adequate separation and retention times are within ±0.05 minutes of previously run standard mixes.

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

5.8.4.5 An acceptable positive and negative control will be stapled together, initialed by the analyst, and placed in the appropriate GC/MS QC notebook. Method conditions should mimic those used in a general screen run by analyst.

Any performance discrepancies or degradation should be reported immediately to a supervisor.

5.8.5 **Tuning the Mass Spectrometer**

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

5.8.5.2 Acceptance criteria for the tune requires evaluation of several areas on the tune print out: *see Tune acceptance criteria chart below.*

Tune Acceptance Criteria (use Atune or Stune)

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

References

Agilent website.
Instrument owner's manual/software.

5.8.5.3 An acceptable tune will be initialed by the analyst and placed in the appropriate GC/MS Tune notebook.

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

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

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

5.8.6 Acceptable results for case work:

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

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

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

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

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

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

5.9 Fourier Transform Infrared Spectrometer (FTIR)

5.9.1 Obtain a background and blank spectrum on the clean accessory. If the accessory exhibits uncharacteristic absorbance, clean the accessory and repeat.

5.9.2 Background and blank spectrums will be printed out and included in the case file.

FTIR Checks

5.9.3 The FTIR will be verified using the Val-Pro provided in the OMNIC software. The instrument should be verified once a week and/or after maintenance has been performed.

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

5.9.5 The Val-Pro report will be printed and kept in the instrument notebook and saved on the instrument computer as the date run.

5.9.6 If any quality control fails to meet acceptable criteria, the bench will be re-aligned and the Val-Pro will be repeated.

Monthly Checks

5.9.7 Bench alignment and quality control checks will be performed in the first working week of use of the month or whenever the bench is moved, modified, powered up, or whenever deemed necessary by the analyst.

5.9.7.1 Turn on the instrument and equilibrate for approximately 30 minutes with the ATR accessory removed and the sampling compartment door closed. Check to ensure that the filter color (green) is acceptable.

5.9.7.2 Create a folder for the monthly check data titled 'mocal_____' using the current date (example mocal060611). Save the folder on the instrument computer.

5.9.7.3 Open the OMNIC software. Align bench and check desiccant.

5.9.7.4 Run the Val-Pro report and print it out. If the Val-Pro report shows a failure, realign the bench and repeat the Val-Pro.

5.9.7.5 Install the ATR accessory and select 'QC' experiment.

5.9.7.6 Collect a background spectrum. Print and save the background spectrum as 'BKG or BKG____' where the blank is the current date.

5.9.7.7 Collect a blank spectrum with nothing on the accessory. Print and save the blank spectrum as 'BLK or BLK_____' where the blank is the current date.

5.9.7.8 Collect a spectrum of the polystyrene card also known as the IR Wavelength Standard using the ATR accessory. Save the spectrum as 'PS____' where the blank is the current date. Insure the serial number and expiration date of the standard is included somewhere on the page.

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

5.9.7.10 Compare the spectra with the guidelines listed below.

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

Acceptable Quality Control Checks

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

5.9.8.1 BKG range approximately 0-5 AU

5.9.8.2 BLK range approximately -0.020 to +0.020

5.9.8.3 PS range: 841 ± 10 cm^{-1}, 905 ± 10 cm^{-1}, 1068 ± 10 cm^{-1}, 1154 ± 10 cm^{-1}, 1600 ± 10 cm^{-1}, 2848 ± 10 cm^{-1}, 3024 ± 10 cm^{-1}, 3059 ± 10 cm^{-1}

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

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

Acceptable results for case work:

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

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

5.10 Gases

5.10.1 It is important that the gases used for instrumentation analysis is of the highest quality.

5.10.2 GC/MS instruments require ultra pure Helium to be used as the carrier gas so as not to interfere in the analysis of items.

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

5.10.4 Any gas cylinder pressure at or below 300 psi will be changed that day.

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

5.11 Peer Review

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

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

The administrative reviewer shall check for accuracy and clarity by reviewing:

- the report for typographical or grammatical errors, misspellings, incorrect dates, omissions, identifying case information errors, or data transfer errors.
- each page of the examination documentation has the unique case identifier(unless stapled together), initials of the analyst, and no improper corrections

- any supporting administrative documentation or case communication contains the unique case identifier (unless stapled together) and the initials of the analyst
- the case file contains the property report, examination documentation, and a report

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

- the examination documentation to ensure the case notes, worksheets, photographs and other data support the conclusions
- the conclusions are reasonable and within the acceptable opinions of peers within the discipline
- manual calculations and data transfers are correct
- appropriate procedures and controls are performed and documented
- general initialing, dating, corrections, and unique case identifiers are correct and appropriate
- worksheets are properly utilized and filled out correctly
- all requested examinations have been performed and addressed on the report or documented by case communication

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

Appendix A- Abbreviations Commonly Used

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

Appendix B - Color Reagents

The following lists some color tests and examples of how they react with various drugs. This list is by no means exhaustive as many color tests cross react with several drugs.

Cobalt Thiocyanate – for cocaine and cocaine base

Preparation:

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

OR

b. Dissolve 5 grams of cobalt thiocyanate in 250 milliliters of distilled water.

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

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

Duquenois-Levine – for marijuana

Preparation:

Reagent 1:

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

OR

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

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

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

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

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

Expires: Made only when needed.

Marquis – for opiates, phenethylamines, and general drug screening

Preparation:

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

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

Results:

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

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

Preparation:

Reagent 1:

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

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

Reagent 2:

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

OR

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

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

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

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

Preparation:

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

Procedure:

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

Results: Lysergic Acid Diethylamide (LSD) = purple

Psilocin, Psilocybin = purplish / bluish

Benzocaine, Procaine = yellowish

Simon's Test

Preparation:

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

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

Procedure:

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

Results:

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

References

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

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

Appendix C - Unit Conversions

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

Appendix D - Hypergeometric Sampling Table

Based on a 95% confidence interval and a 90% success rate.

Sample Population	Number of items to test
1-10	all
11-13	10
14	11
15-16	12
17	13
18	14
19-24	15
25-26	16
27	17
28-35	18
36-37	19
38-46	20
47-48	21
49-58	22
59-77	23
78-88	24
89-118	25
119-178	26
179-298	27
299-939	28
949+	29

Appendix E - Statutory/Trafficking Limits in North Carolina (as of June 1, 2011)

Marijuana

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

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

Hashish (extracted resin of marijuana)

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

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

Possession
Exceeds 7 grams - Class 1 misdemeanor.
Exceeds 21 grams - Class I felony.

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

Cocaine

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

Methamphetamine or amphetamine

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

Heroin (opium or opiate)

Trafficking
4 grams or more, but less than 14 grams

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

MDMA, MDA

Trafficking

By weight

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

By tablet count

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

LSD (Lysergic Acid Diethylamide)

Trafficking

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

4-Methylmethcathinone (Mephedrone)

Trafficking

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

3,4 – Methylenedioxypyrovalerone (MDPV)

Possession

1 gram or less – Class 1 misdemeanor

Trafficking

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

Methaqualone

Trafficking

1,000 but less than 5,000 dosage units
5,000 but less than 10,000 dosage units
10,000 or more dosage units

Appendix F – Commonly used Instrument Methods

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

METHOD NAME	INIT TEMP (in °C)	INIT TIME (min.)	RATE 1 (°/min.)	TEMP 1 (in °C)	HOLD TIME (min.)	RATE 2 (°/min.)	FINAL TEMP (in °C)	FINAL TIME (min.)	TOTAL RUN TIME (min.)	MODE	INLET TEMP (in °C)
SHORT*	255	3.20	75	N/A	N/A	N/A	300	2.50	6.30	SPLIT	255
DRGSCN	100	1.75	25	200	0.25	15	300	5.33	18.00	SPLIT	250
LDRGSCN	100	1.75	25	200	0.25	15	300	10.33	23.00	SPLIT	250
SLEEP	100	999.99	N/A	N/A	N/A	N/A	100	N/A	999.99	SPLIT	255
STEROIDSCN	150	1.0	20	N/A	N/A	N/A	300	10	18.5	SPLIT	250

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

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

Appendix G – Special Procedures

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

Appendix H – Equipment

GC-MS A: GC 6890
MS 5973
Autoinjector 7683
Autosampler 7683
Chemstation E.02.00.493

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

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

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

Direct Inject BAC: GC 6890
Chemstation B.04.01 SP1
GC Headspace: GC 7890
Headspace G1888
Chemstation B.04.02 (98)

FTIR Nicolet 4700 FTIR
OMNIC 7.2

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

References

Sampling
NC v Hayes (7Dec96)
NC v Anderson (3Sep85)
NC v Wilhelm (1Nov82)
NC v Wooten (16Jan74)
NC v Riera (1Mar70)
Journal of Forensic Science (38/4:885, 38/3:641, 37/6:1541, 36/2:350, 35/3:713, 29/2:493)
Uncertainty of Measurement
ASCLD/LAB **–** *International* Standard ISO/IEC 17025:2005(E) 5.4.6 Estimation of Uncertainty of Measurement.
ASCLD/LAB **–** *International* Estimating Uncertainty of Measurement Policy (AL-PD-3008-Ver 2.1 effective date: March 9, 2007).
SWGDRUG – *Measurement Uncertainty for Weight Determinations in Seized Drug Analysis Supplemental Document SD-3*, January 28, 2010.
EURACHEM. *Quantifying Uncertainty in Analytical Measurement* 2007. Available from http://www.measurementuncertainty.org/index.html
The NIST Reference on Constants, Units, and Uncertainty 2009. Available from http://physics.nist.gov/cuu/Uncertainty/index.html

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