

Ada County Sheriff's Office Forensic Lab Drug Chemistry Analytical Method Version 7.0

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1. Scope

- **1.1.** Controlled substances are commonly found in solids, liquids and plant material, and vary in size and concentration.
- **1.2.** Established performance-based methods (guidelines) for the analyses of submitted substances have been developed because of this diversity.
- **1.3.** These procedures allow the individual scientist the flexibility to choose the best methods with which to analyze substances submitted.
- **1.4.** ACSO Forensic lab will perform quantitative analysis on methamphetamine only.

2. References

- 2.1. Ada County Procurement Policy & Procedure Manual
- 2.2. Ada County Sheriff's Office Policy Manual with Procedures
- 2.3. ANAB, ISO/IEC 17025:2017 - Forensic Science Testing and Calibration Laboratories Accreditation Requirements. Document Number AR 3125
- 2.4. ISO/IEC 17025 General requirements for the competence of testing and calibration laboratories, Third Edition 2017-11. Reference Number ISO/IEC 17025:2017 (E).
- 2.5. Joint Committee for Guides in Metrology, International vocabulary of metrology - Basic and general concepts and associated terms (VIM), 3rd edition 2008 version with minor corrections, (JCGM 200:2012). Basic and general concepts and associated terms (VIM) for scientific definitions associated with measurement uncertainty. A copy of this document can be found at www.bipm.org/en/publications/guides/vim.html
- 2.6. Ada County Sheriff's Office Forensic Lab Quality Assurance Manual
- 2.7. Ada County Sheriff's Office Forensic Lab General Forensic Science Training Manual
- 2.8. Ada County Sheriff's Office Forensic Lab Health and Safety Manual
- 2.9. SWGDRUG guidelines
- 2.10. NIST OSAC Subcommittee: Seized Drugs
- 2.11. College/University level Chemistry text books
- 2.12. Operation and maintenance manuals for each analytical instrument

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3. Terms and Definitions

3.1. Accuracy

The extent to which a given measurement agrees with the standard value for that measurement. The closeness of agreement between a test result or measurement result and the true value.

3.2. Aliquot

A sample or portion of a total amount.

3.3. Anabolic steroid

Synthetic substances related to the male sex hormones that promote the growth of skeletal muscle and development of male sexual characteristics (anabolic-androgenic steroids).

3.4. Analytical balance

A class of balance designed to measure small mass in the sub-milligram range.

3.5. Background spectrum

Measures the response of the spectrometer without a sample in place, and is subtracted out.

3.6. Balance

A weighing scale used to measure weight or calculate mass.

3.7. Base peak

The most abundant ion formed in the ionization chamber.

3.8. Binomial distribution

A discrete probability distribution used to model the number of successes in a sample of size n drawn with replacement from a population of size N.

3.9. Certified weight

A precision weight with documentation that it meets a certain standard.

3.10. Clandestine

Secret and concealed, often for illicit reasons.

3.11. Composite

The combining of visually similar evidence items into a single item for analysis.

3.12. Coning and quartering

A method used to reduce the sample size without creating a systematic bias, involves pouring the item so it takes on a conical shape then dividing it into quarters, 2 opposite quarters are removed while the other 2 opposite quarters are combined, these are further reduced down by the same method until an appropriate sample size remains.

3.13. Controlled substance

A listed compound found either in Idaho Statutes Title 37 Chapter 27 or the Code of Federal Regulations Title 21 Part 1308.

3.14. Control sample

A comparison standard for verifying or checking the findings of an experiment. A positive control contains the analyte of choice for the method or test being evaluated. A positive control is used to evaluate a positive result. A negative control does not contain the analyte of choice and is used to verify a negative result.

3.15. Depressant

A substance which depresses the central nervous system. Effects include sedation, muscle relaxation, pleasure, removal of anxiety, and an overall sense of calmness.

3.16. Derivatization

A technique used in chemistry which transforms a chemical compound into a product (the reaction's derivative) of similar chemical structure, called a derivative.

3.17. Diastereomer

One of two stereoisomers that are not mirror images of each other and are nonsuperimposable on one another.

3.18. Distribution

Process of making a product available for use or consumption.

3.19. Drug

Medicine or other substance which has a physiological effect when taken.

3.20. Enantiomer

One of two stereoisomers that are mirror images of each other that are non-superimposable on one another.

3.21. Expanded uncertainty

Multiplying the combined standard uncertainty by a coverage factor, k, to give a level of confidence.

3.22. Gain

How much the detector signal is amplified electronically. It affects the ratio of DC voltage to RF frequency on the mass filter which controls the width of the mass peaks. A higher gain yields narrower peaks (more so at higher masses).

3.23. Hallucinogen

A class of substances which affects the senses and perception of an individual. Sights and sounds are changed or enhanced, sense of time is distorted.

3.24. Hash/Hashish

Collection of the oily, THC-rich resin glands found on all exterior parts of the marijuana plant, except the seeds.

3.25. Hash Oil

A concentration of the oil contained in the resin glands of the marijuana plant.

3.26. Heterogeneous

Non-uniform in composition or character.

3.27. Homogeneous

Uniform in composition or character.

3.28. Hypergeometric distribution

A discrete probability distribution that describes the probability of *k* successes in *n* draws, *without* replacement, from a finite <u>population</u> of size *N* that contains exactly *K* successes, wherein each draw is either a success or a failure.

3.29. Ion source

One of the major components of a Mass Selective Detector (MSD) analyzer, device that creates atomic and molecular ions, either by electron impact (EI) or chemical ionization (CI).

3.30. Instrument blank

A volume of clean solvent that is analyzed on an instrument to ensure that the instrument is working properly and/or that there is no contamination issues associated with the instrument.

3.31. Interferogram

Graph on the FTIR which shows constructive/destructive interference.

3.32. Internal standard

Substance added (in a constant amount) to all samples in an analysis to help compensate for several types of random and systematic errors and to obtain an analyte concentration in a calibration curve.

3.33. Isomer

Molecules that have the same molecular formula but have a different arrangement of the atoms in space.

3.34. Isotope

Atoms of a given element which differ from each other in the number of neutrons in the nucleus, their electronic structure being the same.

3.35. Manufacture

Production, preparation, propagation, compounding, conversion or processing of a controlled substance.

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3.36. Measurement uncertainty

An estimated value, within a specified confidence limit, that depicts a value of variability that can be attributed to the result or test.

3.37. Method blank

An analytical control consisting of all reagents and solvents that is carried through the entire analytical procedure.

3.38. Molecular ion

In the mass spectrum, the heaviest ion (the one with the greatest m/z value).

3.39. Narcotic

Medicinally, a compound which dulls the senses, relieves pain, and produces a slumber. Has been used to refer to all controlled substances at times.

3.40. Narcotic drug

Drug made from opiates and opioids.

3.41. Normal distribution

A bell curve showing values are more likely to fall near the average than further away (Gaussian).

3.42. **Opiate**

A naturally occurring compound isolated from raw opium or a semi-synthetic compound produced from a product contained in raw opium.

3.43. **Opioid**

A fully synthetic compound which has central nervous system properties similar to an opiate.

3.44. Palmate

Arrangement of leaflets, having several lobes (typically 5-7) which radiate from one point.

3.45. Pinnate

Vein arrangement in a leaf with one main vein extending from the base to the tip (midrib) and smaller veins branching off the midrib to the outer edge of the leaf.

3.46. Population

The collection of items under discussion. A population may be real or hypothetical; finite or infinite; homogeneous or heterogeneous.

3.47. Primary standard

A commercially purchased compound that is traceable back to a manufacturer. Its identity is confirmed by verifying its composition through a comparison of FTIR or GCMS with literature, a certificate of analysis or a previously confirmed primary standard.

3.48. Precision

The extent to which a given set of measurements of the same sample agree with their mean.

3.49. Racemic

Mixture that is composed of equal amounts of dextrorotatory and levorotatory forms of the same compound.

3.50. Random sample

A sample chosen from a population without bias, each sample is chosen randomly and entirely by chance, such that each sample has the same probability of being chosen at any stage during the sampling process.

3.51. Reagent

A substance used because of its chemical or biological activity or because it takes part in or brings about a particular chemical or biological reaction.

3.52. Reference material

Material or substance, one or more of whose property values are sufficiently homogenous and well-established to be fit for its intended use in measurement or in examination of nominal properties (i.e. drug standards).

3.53. Relative abundance

Percentage of abundance of other peaks in relation to abundance of the base peak.

3.54. Representative sample

A sample of a statistical population that accurately reflects the members of the entire population.

3.55. Residue

Items which are either too small to be weighed accurately or that which remains after the bulk has been removed.

3.56. Resolution

How close two peaks can be and still be identified as separate peaks.

3.57. Root sum squares

The square root of the sum of the squares of a series of related values.

3.58. Salt

Ionic compound composed of positive ions and negative ions, a crystalline material.

3.59. Sample

An aliquot.

OR if used in a sampling plan;

A unit or a number of units selected from a population. Taking a part of a substance, material or product for testing in order to reach a conclusion, make an inference about,

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and/or report on the whole. Sampling should only be used when there is a reasonable assumption of homogeneity of the whole.

The word "sample" is used throughout this manual and discipline. It is not intended to speak to the whole unless it is used with a sampling plan/procedure. Reports with the wording "the sample contains" do not refer to the whole, unless explicitly stated.

3.60. Sampling plan

For an item that consists of a multi-unit population (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.

3.61. 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 samples(s), and a method to ensure the homogeneity of the larger whole (or to make it so).

3.62. Sample selection

A practice of selecting items to test, or portions of items to test, based on training, experience and competence. When taking an aliquot, there is no assumption about homogeneity.

3.63. Secondary standard

A laboratory produced item or case work item whose composition has been verified by comparison of its Category A instrumental data (e.g., GC/MS, FTIR) to that of a primary standard or a scientifically accepted literature reference.

3.64. Smoothing

Used to improve the appearance of the spectrum obscured by noise.

3.65. Solvent blank

A negative control of any solvent that could potentially be used in an analytical scheme.

3.66. Standard uncertainty

A margin whose size can be thought of as "plus or minus one standard deviation", tells us about the uncertainty of an average.

3.67. Stereoisomers

Molecules that have the same molecular formula and sequence of bonded atoms but differ in the three-dimensional orientations of their atoms in space.

3.68. Stimulant

A substance which stimulates the central nervous system. Effects include an increase in energy, pleasure, satisfactions, or alertness and possibly a decrease in appetite.

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3.69. Tare

Reset the zero of the scale display when an empty container is placed on the weighing platform, in order to display only the weight of the contents of the container.

3.70. Trace amount

Refer to Residue.

3.71. Truncate

To shorten (a number) by dropping one or more digits after the decimal point.

3.72. Unit

One member of a population made up of visually similar evidence items. For example, 1000 bindles are submitted and comprise the population. A unit would be one bindle.

4. General requirements

- **4.1. Impartiality** See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual
- **4.2. Confidentiality** See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual
- 5. **Structural requirements** See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual

6. Resource requirements

- **6.1. General** See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual
- **6.2. Personnel** See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual

6.3. Facilities and environmental conditions

- 6.3.1. Standard laboratory safety protocols should be followed. Refer to the ACSO Forensic Lab Health and Safety Manual.
- 6.3.2. To prevent contamination of personnel and the exhibit, the appropriate equipment should be utilized (e.g., gloves, eye protection, etc.). To minimize cross-contamination, personal protective equipment (PPE) should be worn and changed when necessary.
- 6.3.3. Appropriate eye protection shall be worn whenever a hazard to the eyes exists. This would include chemical exposures and alternate light sources (UV, laser, etc.).
- 6.3.4. Examination utensils (e.g., spatulas, forceps, scissors, measuring devices, etc.) used in processing should be cleaned between items of evidence.
- 6.3.5. When using aerosol type chemicals, proper engineering controls, work practice controls, or PPE must be employed to avoid chemical exposure.
- 6.3.6. The Ultrapure Helium gas tank should be changed out prior to the tank becoming empty to prevent contamination from entering the gas lines.
- 6.3.7. Methods that reference temperatures are all approximate. Temperatures do not have to be recorded or monitored.

6.4. Equipment

- 6.4.1. Only suitable and properly operating equipment shall be employed in the drug chemistry lab.
 - 6.4.1.1. Equipment includes:
 - 6.4.1.1.1. Balances.
 - 6.4.1.1.2. Stereomicroscopes.
 - 6.4.1.1.3. GCMS.
 - 6.4.1.1.4. FTIR.

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- 6.4.1.1.5. Ultraviolet light box.
- 6.4.2. The manufacturer's operation manual and other relevant documentation for each piece of equipment shall be readily available.
- 6.4.3. Each piece of equipment that produces a result that has a significant impact on the final result shall be uniquely identified by its serial number (if applicable) or other unique identifier.
 - 6.4.3.1. Equipment that needs to be uniquely identified include balances, GCMS and FTIR.
- 6.4.4. Equipment performance parameters should be routinely monitored and documented.
 - 6.4.4.1. All maintenance is recorded in a logbook.
 - 6.4.4.2. The many types of maintenance that can be performed on the instrumentation in the laboratory are beyond the scope of this document. General maintenance shall be performed on an "as needed" basis. Refer to the manufacturer's manuals for specific maintenance instructions.

6.4.5. Balances

- 6.4.5.1. An intermediate check shall be performed monthly on all balances, using ASTM Class 2 (or equivalent) weights, to ensure they continue to meet the required specifications. Intermediate checks shall also be performed if the balance has been moved, placed back into service, or purchased.
- 6.4.5.2. Before a check is performed, the pan should be clean and the balance level.
- 6.4.5.3. The weights used shall cover the weighing range of the balance and/or the expected weights of items.
- 6.4.5.4. A log shall be kept of the following:
 - 6.4.5.4.1. Balance ID.
 - 6.4.5.4.2. Analyst initials.
 - 6.4.5.4.3. Date Performed.
 - 6.4.5.4.4. Weights used.
 - 6.4.5.4.5. Recorded weight.
 - 6.4.5.4.6. Whether results are within the tolerance of the balance.

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- 6.4.5.5. Each balance has a tolerance range that shall be used to determine if the scale passes the monthly check. This tolerance range shall be located on the balance check spreadsheet.
- 6.4.5.6. If a balance fails an intermediate check (falls outside the tolerance range), the check is repeated. If the balance still fails, the balance will immediately be taken out of service until it can be recalibrated or repaired. The balance shall clearly be labeled "Out of service". Cases that involve the affected balance will be reviewed and any and all issues will be addressed.

6.4.6. Gas Chromatograph/Mass Spectrometer (GCMS)

- 6.4.6.1. GCMS is an instrument that combines the separation capabilities of Gas Chromatography with the compound selectivity of Mass Spectrometry. This instrument combination is used as a Category A technique in drug chemistry analysis. Separately, the GC and MS may be used as Category B tests.
- 6.4.6.2. Supplies (equipment/reagents/standards)
 - 6.4.6.2.1. Spectroscopic grade solvents or better, to include methanol, chloroform, petroleum ether, acetone, acetonitrile, or other appropriate solvents.
 - 6.4.6.2.2. Sodium carbonate, sodium bicarbonate, sodium hydroxide, or appropriate salt/solution.
 - 6.4.6.2.3. Hydrochloric acid or other acid/acidic solution.
 - 6.4.6.2.4. n-tridecane.
 - 6.4.6.2.5. GCMS vials and caps.
 - 6.4.6.2.6. Ultrapure Helium gas.
- 6.4.6.3. Authenticated drug standards.
 - 6.4.6.3.1. A reference collection of primary and secondary standards that are used for comparisons shall be maintained by the lab.
 - 6.4.6.3.2. Analytical reference materials shall be made by diluting a small aliquot of a primary or secondary standard with an appropriate solvent or by performing a liquid/liquid extraction on the standard.
 - 6.4.6.3.2.1. The name of the standard, manufacturer and lot number (if applicable) shall be documented on the vial.
 - 6.4.6.3.2.2. These standard solutions shall be stored by the GCMS or in the freezer or refrigerator.

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6.4.6.3.3. Primary and secondary standards may continue to be used for comparison purposes as long as they are of sufficient quality to ensure the integrity of the analytical results.

6.4.6.4. Instrument Tune

- 6.4.6.4.1. The MS tuning standard used is perfluorotributylamine (PFTBA) and is added directly to the appropriate storage vial on the mass spectrometer and filled as needed.
- 6.4.6.4.2. The PFTBA is maintained in a sealed and controlled environment within the instrument. Based upon years of laboratory experience, PFTBA has been shown to have an extensive shelf life that may extend well beyond manufacturer expiration dates. Therefore, a specific lot may remain in use beyond the labeled expiration, as long as the instrument tune response appears normal. However, should an instrument fail a tune, one should consider PFTBA as a possible cause of the problem in their troubleshooting protocol.
- 6.4.6.4.3. The MS shall be tuned each day that the instrument is used for casework, using an AUTOTUNE (Atune.U).
 - 6.4.6.4.3.1.An exception to this is when a sequence will not be complete in less than 36 hours. The run may continue uninterrupted and an AUTOTUNE will be run at the end of the sequence.
- 6.4.6.4.4. An AUTOTUNE shall be run after every major maintenance procedure (e.g. source cleaning or column change).
- 6.4.6.4.5. Successful tunes shall be initialed by a drug chemist and kept in a logbook.
- 6.4.6.4.6. Definition of a successful tune (using PFTBA)
 - 6.4.6.4.6.1.Low water and air (18, 28, and 32 amu).
 - 6.4.6.4.6.2.Mass assignments within ± 0.2 AMU of 69, 219, 502.
 - 6.4.6.4.6.3. Peak widths (PW) should be consistent and be 0.6±0.1 (PW50).
 - 6.4.6.4.6.4. 69 or 219 should be the base peak.
 - 6.4.6.4.6.5. Base peak abundance should be ≥350,000 but ≤650,000.
 - 6.4.6.4.6.6.Relative abundances should be anything greater than 30% for 219 and anything higher than 1% for 502.

6.4.6.4.6.7.Isotope mass assignments should be ~ 1 AMU greater than the parent peak and the ratios should be 0.5-1.5% for mass 70, 2-8% for mass 220, and 5-15% for mass 503.

6.4.6.5. Method

- 6.4.6.5.1. A capillary column and temperature program shall be used which is capable of resolving the compound(s) in question.
- 6.4.6.5.2. Samples for GCMS analysis shall be diluted or extracted with solvents.
- 6.4.6.5.3. Once extracted, the sample is not considered evidence and does not need to be treated as such.
- 6.4.6.5.4. If the extract is retained, it shall be sealed inside the evidence packaging and returned with the evidence. This shall be noted on the report.
- 6.4.6.5.5. Sample extracts may be injected manually or via an auto-sampler.

6.4.6.6. Interpretation

- 6.4.6.6.1. Identification of unknowns shall be determined by comparing the overall peak shape, baseline resolution, and MS spectrum of the unknown to the drug standard. In general, peaks should be roughly symmetrical and resolved to baseline. However, it is understood that some compounds and matrices may preclude such criteria; the analyst should be able to articulate why their data still meets quality standards (e.g., certain compounds always tail and some closely eluting peaks "shoulders" can still have readily identifiable mass spectra).
- 6.4.6.6.2. The difference between the GC retention times of the unknown and standard shall not exceed 0.040 minutes. For this purpose, the retention times compared will be those shown in the total ion chromatogram (TIC) window.
- 6.4.6.6.3. Chromatography peaks should be of sufficient abundance to produce an acceptable mass spectrum.
- 6.4.6.4. For a mass spectrum to be acceptable for identification:
 - 6.4.6.6.4.1. Analysis of mass spectra is one of pattern recognition. All comparisons for the purpose of confirmation are made between the reference materials (drug standards) and the unknown spectra.
 - 6.4.6.4.2.Identification of the molecular (parent) ion, if normally present. *Note* Some compounds do not have a molecular ion in their mass spectra.

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- 6.4.6.4.3. The base peak of both the reference spectrum and the sample shall be the same. *Note* Some compounds have several ions that, depending on spectral shifting, may change base ions (e.g., cocaine). In these cases, the base ion of the sample does not have to match that of the standard but does have to be present in significant abundance. The ratios of the relative abundances of the major ions, from the sample, should be similar to those of the standard.
- 6.4.6.6.4.4.All ions with a relative intensity greater than approximately 10% of the base peak in the reference spectrum shall be present in the sample spectrum. For those compounds that do not have three major ions above approximately 10% relative abundance (e.g., methamphetamine, methadone), at least 8 ions from the drug standard should also be present in the sample spectrum.
- 6.4.6.4.5. Any ions present in the sample spectrum but not in the reference spectrum should be reviewed for the presence of contamination or the presence of co-eluting compounds.

6.4.6.7. Controls

- 6.4.6.7.1. Method and instrument blanks shall be utilized to check for contamination or carryover issues. A method blank consists of all solvents and reagents that the sample is exposed to while analyzing. Any appropriate solvent can be used as an instrument blank.
- 6.4.6.7.2. Each day the instrument is used for casework, and prior to casework being performed, an instrument blank (negative control) and an authenticated drug standard (positive control) shall be run.
- 6.4.6.7.3. If the tune passes, no contamination is identified that could impact casework, and the known standard performs like it should, then the instrument is clear for use.
- 6.4.6.7.4. Because this is a known standard (positive control), the presence of stray peaks may indicate breakdown products or contamination. If stray peaks are considered significant or expected peaks are not of sufficient abundance, the problem shall be evaluated. This may require instrument maintenance or the vial to be discarded and a fresh vial used.
- 6.4.6.7.5. A method blank shall be run immediately before each sample. It is considered blank if the compound(s) of interest would not be identified using criteria in 6.4.6.6.
 - 6.4.6.7.5.1.If running a sample for investigative purposes only (see 7.4.4.13), method blanks are not required.

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- 6.4.6.7.6. The presence of extraneous peaks in the blank chromatogram may indicate potential contamination. If a blank has an identifiable compound of interest, then the blank shall be rerun to determine if it is contaminated. If it still contains an analyte(s) of interest, it shall be discarded and a fresh vial used.
 - 6.4.6.7.6.1. The sample shall be rerun immediately after the acceptable blank. (Ensure that no solvents or reagents have been changed prior to making a new method blank. If this has occurred, a new sample shall be re-extracted with the current solvents/reagents).
- 6.4.6.7.7. Once a month, and after maintenance, a test mix, consisting of a mix of common drugs or chemicals, shall be run. The chromatogram shall be compared to the previous month's run for response, separation and identification. Any discrepancies shall be noted and addressed (more maintenance or fresh vial), if necessary. The chromatogram shall be retained.
- 6.4.7. **Fourier Transform Infrared Spectrometer** Infrared spectroscopy is a Category A technique in the analysis of substances. Items can be liquid, solid or vapor, but they shall be relatively pure for purposes of identification.
 - 6.4.7.1. Supplies (equipment/reagents/standards)
 - 6.4.7.1.1. FTIR and corresponding analytical software.
 - 6.4.7.1.2. Drug standards.
 - 6.4.7.2. Method
 - 6.4.7.2.1. Background spectra shall be collected immediately before every sample, under the same conditions as the sample.
 - 6.4.7.2.2. Samples shall be run utilizing the Attenuated Total Reflectance (ATR).
 - 6.4.7.2.3. ATR preparation.
 - 6.4.7.2.3.1.Place sample (solid or liquid) on crystal (i.e. diamond), press (if solid) and analyze.
 - 6.4.7.3. Interpretation
 - 6.4.7.3.1. In order to confirm the presence of an analyte in a sample, the scan of the unknown shall match that of a known standard in the internal library produced by ACSO.
 - 6.4.7.3.2. An internal library shall be made using authenticated drug standards.

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- 6.4.7.3.3. Analysis of questioned samples and standards shall be performed under like conditions (same method).
- 6.4.7.3.4. An automatic baseline correction may be applied to each spectrum.
- 6.4.7.3.5. FTIR spectra are considered a match if the peaks of the standard are present in the unknown spectrum, in location, shape and relative intensities. Any extra major peaks in the unknown spectrum must be explainable.
- 6.4.7.3.6. If spectral subtraction is done, then what was subtracted shall be in the case notes.
- 6.4.7.3.7. If an FTIR spectrum is inconclusive or negative for a controlled substance, the item shall then be analyzed on a GCMS.

6.4.7.4. Controls

- 6.4.7.4.1. Perform a laser verification and align the laser (bench), in that order, once a month. If the instrument is moved or had maintenance, perform both of these procedures before utilizing. Documentation shall be saved.
- 6.4.7.4.2. An instrument performance verification and an advanced diagnostics test shall be run monthly and after any maintenance. Using the manufacturer's procedures, a performance verification of the instrument is done using a traceable polystyrene film. Ensure everything passes. Documentation shall be saved.
 - 6.4.7.4.2.1. The traceable polystyrene film (both an internal "wheel" and an external "lollipop") may remain in use beyond the labeled expiration, as long as the instrument verification response appears normal. However, should an instrument fail a verification, one should consider the wheel, lollipop, or both as a possible cause of the problem in their troubleshooting protocol.
 - 6.4.7.4.2.2.If the verification does not pass and/or there is any other symptom of system failure, perform another laser verification and align the laser (bench) and/or consult the manufacturer.
- 6.4.7.4.3. Check the desiccant monthly and replace if necessary.

6.5. Metrological traceability

6.5.1. Weights shall be NIST traceable and certified at time of purchase. The documentation for the certification/calibration of the weights shall be retained.

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- 6.5.2. Weights used to check the balance accuracy shall be re-certified every accreditation cycle by an ISO/IEC 17025 accredited vendor whose scope of accreditation covers the calibration performed.
- 6.5.3. Annually, balances shall be calibrated by an outside vendor that is accredited to ISO/IEC 17025 and whose scope of accreditation covers the calibration performed.

6.6. Externally provided products and services

- 6.6.1. The following supplies/services influence the quality of tests and must meet the listed requirements:
 - 6.6.1.1. Gas for the GCMS shall be ultrapure helium.
 - 6.6.1.2. All solvents used shall be spectroscopic grade or better.
 - 6.6.1.2.1. Chloroform shall be stabilized with Ethanol or Pentene.
 - 6.6.1.3. Balance, weight, and pipette calibrations shall be performed by an ISO accredited laboratory.
 - 6.6.1.4. Reference materials shall be purchased from an accredited supplier, if available.
 - 6.6.1.5. Test reagents shall not be used until they have been verified by passing a quality control test. The test shall be documented.

7. Process requirements

- **7.1. Review of requests, tenders, and contracts** See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual
- **7.2. Selection, verification, and validation of methods** See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual

7.3. Sampling

- 7.3.1. The principal purpose of sampling is to allow the analyst to draw a conclusion about the entire population by examining only a portion of the population (i.e., a sample). If a scientist is only analyzing a single item or drawing a conclusion regarding only the items tested with no inference to the entire population, then a sampling plan is not needed. If no sampling plan is needed, then analysis can be accomplished in one of two ways:
 - 7.3.1.1. Itemize the examined unit (or multiple units) separately from the non-examined units. The report would then have a finding associated with the analyzed unit(s) and a "Not analyzed" finding associated with the non-examined unit(s).

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- 7.3.1.2. Keep the population whole (i.e. not itemized out), but fully examine one unit. The report would then clearly convey that the finding is attributed to only one unit and the rest of the units were not analyzed.
 - 7.3.1.2.1. Example: "20 white oblong tablets with imprint M367", analyzed one. The sample contains Hydrocodone (CII)."
- 7.3.1.3. With a statistical sampling plan the analyst can test a sample of a visually-consistent population and report a result that applies to the entire population with a stated level of confidence. The statistical sampling plan utilized shall be the hypergeometric sampling plan. Refer to the Hypergeometric Table (Table 1). This table was constructed using the hypergeometric calculation based on a 95% probability that 90% of the population contains the identified compound. For an indepth explanation of this calculation, click on the link provided, https://www.unodc.org/documents/scientific/Drug Sampling.pdf.
 - 7.3.1.3.1. The case notes shall clearly indicate if a hypergeometric sampling plan was used. The sampling plan used does not need to be included in the report.
 - 7.3.1.3.2. An observation shall be conducted to determine that one homogenous population is present. If more than one population is present, the sampling plan shall be used on each type of population.
 - 7.3.1.3.3. The analyst shall include in their notes the population size and the number to be tested.
 - 7.3.1.3.3.1. Example: Hypergeometric Sampling Plan, Population size = 40, Number of samples to be tested = 20.
 - 7.3.1.3.4. The appropriate number of specimens within the population will be randomly selected.
 - 7.3.1.3.5. Date and time of sampling are not relevant and do not need to be documented. Sampling location does not need to be identified nor is there any environmental or transport conditions that affect the interpretation of the test results.
 - 7.3.1.3.6. The report shall indicate the corresponding confidence limits (i.e., "95% confident that at least 90% of the items in the population contain the reported drug").

Table 1: Hypergeometric Table

Total number of items in exhibit	Number of units to be tested	
1-10	All units	
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	
940+	29	

7.4. Handling of test or calibration items

7.4.1. Physical Examination

- 7.4.1.1. After removing the item from its packaging (if appropriate), a thorough physical characterization of the item shall be conducted and the following observations should be noted if applicable: type of material, color, size, shape, amount, morphology, significant markings, odor and texture.
 - 7.4.1.1.1 Pills/tablets shall have the entire imprint/logo documented. Documentation can be written, sketched, photographed, or photocopied.
 - 7.4.1.1.2. Any photographs taken of drug evidence is for documentary purposes only. Any file format is acceptable. The photographs may be archived in the note packet or may be uploaded to the agency's record management system.

- 7.4.2. Weighing
 - 7.4.2.1. At some point during the physical examination of the item, a weight or count must be determined. Prior to weighing an item, the analyst shall make sure the balance is clean and tared.
 - 7.4.2.1.1. Exceptions to this include when dealing with residue amounts or an item whose nature precludes an accurate weighing (sticky, sludge, moldy, etc.).
 - 7.4.2.1.2. The reason why it was not weighed must be in the case notes.
 - 7.4.2.2. Residue or trace shall be defined as anything less than 0.10 grams.
 - 7.4.2.3. Items that contain a small amount of material that clings to the packaging can be considered residue and reported as such.
 - 7.4.2.3.1. Smoking devices that contain a substance packed into the bowl (regardless of the amount) do not have to be removed or weighed and can be reported as residue.
 - 7.4.2.4. Net weights are determined by weighing only the contents of a container.
 - 7.4.2.5. Gross weight measurements are made by weighing the entire item (packaging and contents).
 - 7.4.2.6. All digits observed from the balance shall be recorded in the case notes.
 - 7.4.2.7. When the total weight of an item(s) falls within the window of uncertainty at regulatory limits, then the uncertainty associated with each weighing event must be listed on the report. See Section 7.6.3.
- 7.4.3. Categorizing Analytical Techniques
 - 7.4.3.1. Techniques for the analysis of drug samples are classified into three categories (see Table 2) based on their maximum potential discriminating power. However, the classification of a technique may be lower, if the item, compound, or mode of operation diminishes its discriminating power. An example of diminished discriminating power is an infrared spectroscopy technique applied to a mixture which produces a combined spectrum.

Table 2: Categories of Analytical Techniques

Category A	Category B	Category C
Fourier Transform Infrared Spectroscopy (FTIR)	Gas Chromatography (GC)	Color Tests
GCMS	Mass Spectrometry (MS)	Ultraviolet (UV) Light box
	Thin Layer Chromatography (TLC)	
	Pharmaceutical Identifiers	
	Cannabis only:	
	Macroscopic Examination	
	Microscopic Examination	

7.4.4. Identification Criteria

- 7.4.4.1. 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, if possible. Category A is considered a confirmatory test since it gives structural information about the drug sample. Category B and C are considered presumptive/preliminary tests.
- 7.4.4.2. When a Category A technique is not used, at least three different validated techniques shall be employed from Category B and/or C.
- 7.4.4.3. This combination shall identify the specific drug present.
- 7.4.4.4. For quality assurance purposes, a second sample shall be taken and a second technique shall be applied to it, when sample size allows for it. Case notes shall specify when multiple techniques are applied to the same sample and why.
- 7.4.4.5. If only one sample selection can be performed on a sample, then an n-tridecane internal standard (IS) shall be added to the extract before analysis on the GCMS. A blank with internal standard shall be run prior to the extract.
 - 7.4.4.5.1. Add a couple drops of the IS (1.3mL of n-tridecane to 1L of chloroform) to the sample and blank extract vials prior to analysis.
 - 7.4.4.5.2. Caution, the IS is volatile. Care must be taken when analyzing the sample extract to ensure there is an adequate amount of the IS for detection.

- 7.4.4.6. In order to report "No Controlled Substance(s) detected", at a minimum, a sample must be run on the GCMS using a temperature program and extraction scheme that encompasses a wide range of drugs.
- 7.4.4.7. A sample extract shall be defined as any casework related extract, solution, or solid that is not returned to evidence.
- 7.4.4.8. If a compound is derivatized as part of the analytical scheme, the GCMS of the non-derivatized sample in combination with the GCMS for the derivatized sample is considered two separate tests in Category A, provided that they are from two separate sample selections as described above.
- 7.4.4.9. Confirmatory data from Category A techniques (Table 2) shall be compared to an authenticated drug standard. On the GCMS, the standard shall be run within 24 hours of running the unknown. For the FTIR, the standard shall be in the internal library produced by ACSO.
- 7.4.4.10. All analytical techniques shall have documentation that is reviewable. Examples of reviewable documentation include:
 - 7.4.4.10.1. Spectra, chromatograms, and photographs/photocopies of TLC plates.
 - 7.4.4.10.2. Pharmaceutical identifiers (i.e. notes shall include Drugs.com, Drug ID Bible or other identifier page(s)).
 - 7.4.4.10.3. Documentation in notes (i.e. color results, microscopic/macroscopic results).
- 7.4.4.11. For any method to be considered of value, the test must 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 forensic identification of a drug.
- 7.4.4.12. If an analyst has met or exceeded the requirements for the identification of a substance, the report shall simply state the result, and the Schedule (if applicable) in a section easily identifiable as "Opinions", "Interpretations", or similar wording.
- 7.4.4.13. For investigative purposes, an item can be hand delivered to the laboratory and analyzed with color tests and/or on the GCMS or FTIR. If instrumentation is utilized, the result shall be compared to a library search. This analysis will be documented in case notes.
 - 7.4.4.13.1. If confirmation is required, a sample may be retained to perform further testing.

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7.4.4.13.2. A report shall be issued for any analysis performed in the laboratory. If a confirmation test is not run, the report shall clearly state that the results are preliminary.

7.4.5. Minimum Examination Requirements

- 7.4.5.1. All controlled substances charged as felonies, in the State of Idaho, should be confirmed, if possible. **Exceptions include**: inadequate sample size, inability to obtain a standard, and compounds that are of the same drug class that have an instrumental response that is relatively minor compared to the major peak and/or can reasonably be assumed to be a byproduct of the manufacturing process. Examples include, but are not limited to, morphine and codeine in a heroin sample or P2P from a suspected clan lab.
- 7.4.5.2. If a case has a mixture of compounds from different schedules, it is up to the analyst to determine what warrants confirmation. If it does not, then the report shall note that the sample indicates the presence of another controlled substance that was not confirmed.
- 7.4.5.3. All controlled substances should be scheduled, if possible. **Exception**: liquid item in unmarked bottle containing a controlled substance, where the schedule is dependent on the concentration of that controlled substance. If liquid item comes in a labeled pharmacy bottle and the results of analytical testing confirm the presence of the ingredients on the label, then the schedule on the label shall be reported.
- 7.4.5.4. Reporting of non-controlled substances shall be left up to the discretion of the analyst.
- 7.4.5.5. If control samples (solvent blanks) are submitted with syringe washes, they shall be treated as regular samples.

7.4.6. General Information

- 7.4.6.1. Attempts shall be made to preserve evidence. The analyst shall make an effort to ensure enough of the evidence remains after analysis to allow for reexamination.
- 7.4.6.2. When only a trace level of an item is present, every effort shall be made to use less than one half of the item. If it is necessary to use the entire item, then any extracts, left over liquids, or residues shall be returned to the evidence envelope.
- 7.4.6.3. Any extracts (washes) from evidence that did not have any visible residue shall be returned with the evidence.
- 7.4.6.4. Laboratory notes shall contain the following, if applicable:

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- 7.4.6.4.1. A description of the packaging (plastic bag, glass vial, bindle, etc.).
- 7.4.6.4.2. A description of the item (powder, liquid, plant material, tablet including color, shape and imprint).
- 7.4.6.4.3. Weight or count (tablets/capsules) of item.
- 7.4.6.4.4. Balance used for weighing.
- 7.4.6.4.5. Reserve weight or count.
- 7.4.6.5. GCMS spectra shall include:
 - 7.4.6.5.1. Instrument name.
 - 7.4.6.5.2. Date of analysis.
 - 7.4.6.5.3. Method utilized.
 - 7.4.6.5.4. Case number, exhibit number and analyst's initials or manufacturer and/or lot number of the drug standard.
- 7.4.6.6. FTIR spectra shall include:
 - 7.4.6.6.1. Instrument name.
 - 7.4.6.6.2. Date of analysis.
 - 7.4.6.6.3. Operating parameters.
 - 7.4.6.6.4. Case number, exhibit number and analyst's initials.
 - 7.4.6.6.5. Library match from ACSO library.
- 7.4.6.7. In multi item exhibits, the report shall clearly state what, and how many, items were tested. Example: "Three white oval tablets with imprint "M367", analyzed one."
- 7.4.6.8. If an analytical scheme is employed that is restricted to a limited range of compounds, that limitation shall be clearly stated on the report along with any qualifiers. Example: "No basic drugs detected, examples of basic drugs include opiates, amphetamines, and cocaine."
- 7.4.7. Sample Selection Rules
 - 7.4.7.1. Sample selection rules allow for the analysis of key evidence items within

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a case to maximize the resources of the lab. Requests for analysis on items not originally tested shall be reviewed and prioritized on a case by case basis.

- 7.4.7.2. A felony charge has priority over a misdemeanor. Example: a gram of cocaine found in a suspect's pocket shall be tested while a gram of marijuana found in the same pocket may not be.
- 7.4.7.3. A misdemeanor is treated equally to a felony if it is closer to the suspect or was the probable cause for a subsequent search. Example: A gram of marijuana found in a suspect's pocket would be analyzed in addition to a gram of cocaine found in the suspect's car.
- 7.4.7.4. Based on the analyst's training and experience, if it is suspected that different types of felony drugs are submitted then one of each type shall be analyzed. The analyst may use resources such as: statements of fact, description of items as well as visual inspection of items in making this determination.
- 7.4.7.5. The analyst shall always strive to provide evidence supporting the highest charge (i.e. trafficking, manufacturing, delivery vs. felony possession vs. misdemeanor possession).
- 7.4.7.6. For less than trafficking amounts, the number of items necessary to support the charge shall be analyzed. Example: If you have five items and the charge is possession, then only one item needs to be tested. If the charge is intent to deliver, then more items may need to be tested. Consultation with the prosecutor and/or officer should determine the number needed. The report shall state the total number of items, the weight of the number actually analyzed, and the findings.
- 7.4.7.7. For trafficking amounts, **ALL** items shall be analyzed until the appropriate trafficking level is reached. Example: Forty balloons are submitted, each with about 0.1g of suspected heroin, the analyst shall continue to test balloons until they reach the first trafficking level, 2.0g.

7.4.8. Pharmaceuticals

- 7.4.8.1. Pills that have recognizable logos and/or identification numbers need analytical confirmation if a literature search indicates that they contain a Schedule II controlled substance. Exception: if a controlled substance has been analytically confirmed from a non-pill item in the case, then a pill(s) listed to contain the same controlled substance only needs a literature search. If a literature search reveals that pills with two, or more, different labels contain the same controlled substance, then only one of the pills needs to be analyzed. For the purpose of satisfying the identification criteria, the search shall be considered a presumptive test.
 - 7.4.8.1.1. While pharmaceutical identifiers are typically a reliable indicator of the content of a tablet, there are known instances of counterfeit tablets and errors

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in reference material. Analysts should take care to observe and note any abnormalities in color, size or markings that may reveal that the item is not a legitimate product. If a counterfeit is suspected or discovered, the evidence is no longer treated as a pharmaceutical in nature.

- Acceptable literature references are published books (e.g. PDR, DIB, Logo index etc.), manufacturer's websites, Drugs.com, and labels from pharmaceutical packaging.
- 7.4.8.3. All literature searches shall have the source documented in the case notes.
- Information from poison control centers and non-manufacturers' 7.4.8.4. websites can be used as a preliminary test (Table 2, Category B pharmaceutical identifier) when further analytical testing is performed or in conjunction with published books or approved websites to delineate pills with similar imprints and descriptions.
- 7.4.8.5. Sealed pharmaceutical packaging (i.e. manufacture-sealed boxes, blister packs, and foil pouches) may be used as a Category B test (Table 2) if there is no indication of tampering.
- When analyzing a pharmaceutical tablet where some portion of the tablet or manufacturer's logo is missing, care should be taken when determining whether the remaining logo portion is sufficient to use as a Category B test (Table 2). Generally, a partial logo should not be used as a presumptive test (Category B). However, a logo may be sufficiently unique that the remaining logo portion is still readily identifiable. In those cases, the partial logo may be used as a presumptive identifier (Category B). Analysts should record other physical characteristics of the tablet that could assist in its identification (e.g. color, shape, dimensions, etc.).
- 7.4.8.7. A sample from each type of two-part, unsealed, gelatin type capsules shall be analyzed.

7.4.9. Color Tests

Chemical color tests produce characteristic color reactions that provide information regarding the nature of the substance being tested. Certain compounds or classes of compounds produce distinct colors when brought into contact with various chemical reagents. These simple reactions can indicate the presence of a particular functional group or molecular moiety. Colors are subjective and can be influenced by the concentration of the sample, the presence of contaminants, or by the age of the reagent.

- 7.4.9.1. Supplies (equipment/reagents/standards)
 - 7.4.9.1.1. Spot plates, test tubes, spatulas.

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7.4.9.1.2. Color test reagents, sulfuric acid, chloroform (commonly stored in dropper bottles), UV light box.

7.4.9.2. Reagent preparation

7.4.9.2.1. Directions for preparation of the commonly used color test reagents and the results can be located in <u>Clarke's Analysis of Drugs and Poisons</u> or other appropriate sources, to include an in-house log of color test results. The reagent log shall document the recipe used.

7.4.9.3. Method

- 7.4.9.3.1. Place 1-2 drops of reagent in a depression of a spot plate. Ensure no color develops.
- 7.4.9.3.2. Add a small amount of sample.
- 7.4.9.3.3. Observe and record results.
- 7.4.9.3.4. Some tests require the use of a test tube instead of a spot plate (modified Duquenois-Levine or Scott's test).

7.4.9.4. Interpretations

7.4.9.4.1. Color test results shall be recorded in the case notes by detailing the color or lack of color reaction that results. If other reactions besides color are observed, they should also be recorded (i.e. effervescence, fuming, precipitation).

7.4.9.5. Cautions

- 7.4.9.5.1. If there is reason to suspect that the reagent is old or no longer working properly, it should be tested with a substance which will react with the reagent to produce a color. If the reagent does not produce the expected color with this substance, the reagent shall be discarded.
- 7.4.9.5.2. Most color tests work relatively quickly, depending on the concentration of the sample. Be aware that many color reagents are made with a strong acid that will eventually char/burn the sample and appear brown/black in color.

7.4.9.6. Controls

7.4.9.6.1. A blank (negative control) shall be run before each test. Place the appropriate reagent in a blank well and observe reaction (or lack thereof) prior to placing sample into well. Ensure no reaction occurs prior to proceeding with addition of the sample. Refer to 7.7.1.2.

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7.4.9.6.2. A positive control shall be tested when a reagent is made (and monthly, if spelled out in 7.7.1.4.1).

7.4.9.6.3. The results of one time use controls shall be documented in the case notes.

7.4.10. Thin Layer Chromatography (TLC)

7.4.10.1. Supplies (equipment/reagents/standards)

7.4.10.1.1. Thin layer chromatography tank and silica gel plates.

7.4.10.1.2. Aerosol spray units, capillary tubes, test tubes.

7.4.10.1.3. Developing sprays, solvents (many different types may be employed depending upon the compound of interest).

7.4.10.2. Methods

- 7.4.10.2.1. A pencil-drawn line is placed on the TLC plate approximately 1-2 cm from the bottom of the plate, creating an origin line. The sample(s) and standard(s) are spotted on this line, allowing adequate space between the sides of the plate and between the samples themselves such that the samples do not migrate into each other's paths or off the edge of the plate (1 cm is ideal). A capillary tube is used for spotting. A small, tight spot is important for best results. The spotting solvent shall be volatile and relatively non-polar to minimize the wandering of the initial spot. The spot shall be dried (evaporated or heat dried) prior to entering the mobile phase of the test. Appropriately label the plate before spotting.
- 7.4.10.2.2. A developing chamber utilizing the glass tank apparatus and appropriate solvent(s) should be assembled and allowed to equilibrate before adding the spotted plate. The solvent of choice for the mobile phase will vary depending upon the compounds of interest. If the target compound or group of compounds is known, a good reference book (e.g. Clarke) will outline a TLC system that works for the compound.
- 7.4.10.2.3. Place the spotted TLC plate into the chamber in a vertical position with the origin line at the bottom but above the mobile phase solvent. The solvent should be allowed to migrate up the length of the plate. Attempt to remove the plate prior to the solvent reaching the top; spots may be lost if plate stays in the tank too long. A shorter migration distance increases sensitivity (the sample is concentrated) but decreases resolution. After the solvent has traveled the required distance, the plate is removed. The plate is allowed to dry by evaporation or heated with warm air. The plate is placed in a ventilated area and sprayed with an appropriate visualizing compound (depending upon the compounds screened for). Any developed spots are circled immediately if they are not clearly visible.

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7.4.10.3. Interpretations

- 7.4.10.3.1. Compare results of unknown to those of standard. A spot shall match color and location (in-line with) of the standard for a positive preliminary result. The line of spots across the TLC plate should form a continuum, be it linear or parabolic.
- 7.4.10.3.2. Document the plate for the case file.

7.4.10.4. Controls

- 7.4.10.4.1. A blank and an authenticated drug standard(s) shall be run with each plate.
 - 7.4.10.4.1.1. A blank TLC shall have no spot in the "blank" column.
- 7.4.10.4.2. The results of the controls shall be documented in the case notes.

7.4.11.Marijuana

This procedure is composed of a series of tests, none of which by themselves are specific for marijuana or THC, but taken in combination, are considered specific for the presence of marijuana or its resins.

- 7.4.11.1. Supplies (equipment/reagents/standards)
 - 7.4.11.1.1. Stereomicroscope.
 - 7.4.11.1.2. Thin layer chromatography tank and silica gel plates.
 - 7.4.11.1.3. Aerosol spray units, capillary tubes, testtubes.
 - 7.4.11.1.4. Aqueous Fast Blue BB solution, or other appropriate visualizing spray.
 - 7.4.11.1.5. Petroleum ether, methanol, chloroform (stabilized with ethanol or pentene), ethyl acetate, acetonitrile, ether or other appropriate solvent.

7.4.11.2. Method

7.4.11.2.1. Plant material is first examined macroscopically for the following characteristics: palmate arrangement of leaflets, pinnate leaf venation, serrated edges of the leaflets or leaf fragments, buds, fluted stems and stalks, seeds, stems, and flowers consistent with marijuana.

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- 7.4.11.2.2. Plant material is then examined using a stereomicroscope for the following characteristics:
 - 7.4.11.2.2.1. Cystolithic hairs-Small "bear claw" shaped hairs with bases of calcium carbonate. The cystolithic hairs are located on the topside of the leaf or leaf-fragment.
 - 7.4.11.2.2.2. Unicellular hairs- Fine hairs located on the underside of theleaf or leaf-fragment. Note: Unicellular hairs are not always observed on the leaves from the budding parts of the marijuana plant.
 - 7.4.11.2.2.3. Seeds are examined using a stereomicroscope for the following characteristics:
 - Veined shell.
 - Ridged edges.
 - Point on one end and dint on the end of plant attachment.
- 7.4.11.2.3. Place plant material/residue/sample in test tube. The modified Duquenois-Levine test may be performed directly on the plant material/residue/sample. If an extract is needed, cover with appropriate solvent, or flush smoking device with appropriate solvent if no plant material observed or unable to distinguish macroscopically and/or microscopically.
 - 7.4.11.2.3.1. Use this extract for modified Duquenois-Levine or thin layer chromatography, if necessary.
- 7.4.11.2.4. Retain a small amount of unused solvent as a blank.
- 7.4.11.2.5. Modified Duquenois-Levine test
 - 7.4.11.2.5.1. In a test tube containing plant material/residue/sample or a portion of the evaporated solvent extract, mix 2-10 drops of Duquenois reagent and an equal amount of concentrated HCl.
 - 7.4.11.2.5.2. Let stand $\frac{1}{2}$ to 3 minutes and observe a color change.
 - 7.4.11.2.5.3. Add chloroform.
 - 7.4.11.2.5.4. Observe if purple color transfers into chloroform layer.
 - 7.4.11.2.5.5. Record results in the case file.
- 7.4.11.2.6. Thin Layer Chromatography
 - 7.4.11.2.6.1. Spot a small amount of solvent extract onto a TLC plate alongside of a marijuana standard and solvent blank.
 - 7.4.11.2.6.2. Develop the plate using one or more of the following

mobile phases:

- Chloroform
- Ethyl Acetate
- 4:1 Petroleum Ether/Ether
- 7.4.11.2.6.3. Visualize by spraying the plate with Fast Blue BB salt solution or other acceptable visualizing spray.
- 7.4.11.2.6.4. Compare results of unknown to those of standard. Document the plate for the case file.
- 7.4.11.2.7. Marijuana may also be confirmed on the GCMS, as long as the identification criteria are met per 7.4.4.1 and/or 7.4.4.5.
- 7.4.11.2.8. Marijuana shall be reported using the words "marijuana or the resins thereof".

7.4.11.3. Interpretations

- 7.4.11.3.1. A positive macroscopic test shall be defined as any combination of at least two of the characteristics listed in 7.4.11.2.1.
- 7.4.11.3.2. A positive microscopic test shall be defined as an observation of cystolithic hairs and unicellular hairs (unicellular hairs may not be present on the buds) on the leaf and/or the presence of characteristic seeds.
- 7.4.11.3.3. A positive Thin Layer Chromatography test shall be defined as the presence of a red spot with migration distance consistent with the red THC spot of the standard. The line of spots across the TLC plate should form a continuum, be it linear or parabolic. The blank shall be negative (no spot).
- 7.4.11.3.4. A positive Modified Duquenois-Levine test shall be defined as a purple color developing after the addition of the HCl (color may vary from blue to reddish purple depending on the sample) and transfer of the purple color into the organic layer after the addition of chloroform.
- 7.4.11.3.5. To report the sample contains marijuana or the resins thereof, you shall have one of the following:
 - 7.4.11.3.5.1. Positive macroscopic, positive microscopic and a positive modified Duquenois-Levine.
 - 7.4.11.3.5.2. Positive microscopic, positive single TLC system and positive modified Duquenois-Levine.
 - 7.4.11.3.5.3. Negative microscope, positive modified Duquenois-Levine and two positive TLC systems.

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7.4.11.3.5.4. GCMS results that match to a marijuana standard with at least one other positive technique from Category A, B, or C (if not possible, see 7.4.4.5).

7.4.11.4. Controls

- 7.4.11.4.1. A blank and a standard shall be run with each batch of TLC plate(s). The blank (negative control) is the extraction solvent. The standard (positive control) is an authenticated marijuana standard.
- 7.4.11.4.2. The results of the controls shall be documented in the case notes.

7.4.12.Psilocybin Mushrooms

- 7.4.12.1. Supplies (equipment/reagents/standards)
 - 7.4.12.1.1. GCMS and appropriate analytical software.
 - 7.4.12.1.2. TLC equipment.
 - 7.4.12.1.3. Methanol, acetone, or other appropriate solvents.
 - 7.4.12.1.4. Fast Blue BB, p-Dimethylaminobenzaldehyde (pDMAB), or other appropriate solvents and/or visualizing sprays.
 - 7.4.12.1.5. Ammonium hydroxide (NH4OH)
 - 7.4.12.1.6. Spot plates, pipettes, test tube caps.
 - 7.4.12.1.7. Freezer.

7.4.12.2. Method

- 7.4.12.2.1. Weber Color Test
 - 7.4.12.2.1.1. Add Fast Blue BB to spot plate. Ensure no color develops. Add sample. A positive test will turn red-orange within a couple of minutes.
 - 7.4.12.2.1.2. Add a drop of concentrated HCl. You can also transfer some of the liquid to another clean well prior to adding a drop of concentrated HCl. A positive test will turn a blue-green color.
- 7.4.12.2.2. GCMS Sample Preparation and Analysis
 - 7.4.12.2.2.1. There are multiple extractions that can be performed on mushrooms to determine if they contain psilocybin and/or psilocin. The

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matrix of the item may determine the appropriate extraction to use. Listed below are a couple of extraction procedures. However, it is up to the analyst's discretion to determine the appropriate extraction method to use based on their training and experience with different types of extractions.

- 7.4.12.2.2.2. Extract with just enough methanol to cover the sample in a test tube. At this stage, the extract can be injected into the GCMS.
- 7.4.12.2.2.3. Centrifuge and decant solution into a clean test tube. Cap and place into freezer for at least one hour.
- 7.4.12.2.2.4. Remove from freezer and immediately add an equal volumeof acetone, mix.
- 7.4.12.2.2.5. Centrifuge, decant, and if necessary, concentrate the supernatant.
- 7.4.12.2.2.6. Run on GCMS, using a split or splitless data acquisition method depending on the sensitivity of the instrument.
- 7.4.12.2.3. Thin Layer Chromatography
 - 7.4.12.2.3.1. If differentiation of psilocin and psilocybin is required, run a T1 system (10 mL methanol + 7 drops of ammonium hydroxide (NH4OH)), developed with pDMAB.

7.4.12.3. Interpretations

- 7.4.12.3.1. Compare the results with an authenticated psilocin or psilocybin standard. Psilocybin breaks down into psilocin in the hot injection port of the GC.
- 7.4.12.3.2. Positive results shall be reported as "psilocin and/orpsilocybin". If differentiated with TLC, then the compound identified will be reported.

7.4.12.4. Controls

- 7.4.12.4.1. Negative and positive controls shall be run with the color tests.
 - 7.4.12.4.1.1. An authenticated psilocin/psilocybin standard shall be used as a positive control.
 - 7.4.12.4.1.2. A clean well shall be used as a negative control.
- 7.4.12.4.2. The results of the controls shall be documented in the case notes.

7.4.13.LSD

- 7.4.13.1. Supplies (equipment/reagents/standards)
 - 7.4.13.1.1. GCMS and appropriate analytical software.
 - 7.4.13.1.2. Ultraviolet (UV) light source.
 - 7.4.13.1.3. pDMAB color reagent.
 - 7.4.13.1.4. TLC equipment.
 - 7.4.13.1.5. Methanol, chloroform, or other appropriate solvents.
 - 7.4.13.1.6. Sodium carbonate, sodium bicarbonate, sodium hydroxide, or appropriate basic salt/solution.
- 7.4.13.2. Method
 - 7.4.13.2.1. Preliminary tests.
 - 7.4.13.2.1.1. Place evidence under the UV light. LSD exhibits a blueviolet fluorescence in the presence of ultraviolet light.
 - 7.4.13.2.1.2. It is common for white paper to reflect the UV and appear blueviolet even without LSD. If the entire sample fluoresces (no differentiation from background and suspected location of LSD), then a methanol extract can be performed and then observed under UV light.
 - 7.4.13.2.1.3. In addition to, or in place of the UV light test, add pDMAB to a spot well. Ensure no color develops. Add sample. A positive test will turn purple.
 - 7.4.13.2.1.4. TLC can also be used as a preliminary test to identify LSD. A good reference book (e.g. Clarke) will outline a TLC system and visualization spray that works for LSD. A TAE system (methanol), developed with Van Urk's reagent, and then heated in oven at approximately 100°C for 5 min, will produce a blue spot for LSD.
 - 7.4.13.2.2. GCMS Sample Preparation and Analysis
 - 7.4.13.2.2.1. "Window panes", blotter paper and pulverized microdots can be extracted directly with methanol. Place sample in test tube and add just enough methanol to cover the sample. Shake and let soak. Time can vary but they may need to soak for at least an hour. Centrifuge, if necessary, and analyze.
 - 7.4.13.2.2.2. Sugar cubes, "SweeTarts" or other candy. View item under UV to find the area of suspected LSD. Scrape off upper layer

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(attempt to reserve half of the item). Dissolve sample in water and make basic. Extract with chloroform and run on the GCMS.

7.4.13.2.2.3. The GCMS should be set to splitless mode. The liner may have to be changed to a splitless liner depending on the sensitivity of the instrument.

7.4.13.3. Interpretations

- 7.4.13.3.1. Compare the results with an authenticated LSD standard.
- 7.4.13.3.2. Positive results shall be reported as "lysergic acid diethylamide (LSD, Schedule I)."

7.4.13.4. Cautions

- 7.4.13.4.1. The retention time is concentration dependent.
- 7.4.13.4.2. LSD degrades in the presence of light and/or elevated temperatures.

7.4.13.5. Controls

- 7.4.13.5.1. If a color test is utilized, a negative and positive control shall be run and documented in the case notes.
 - 7.4.13.5.1.1. An authenticated LSD standard shall be used as a positive control. A purple color will develop if using pDMAB.
 - 7.4.13.5.1.2. A clean well shall be used as a negative control.
- 7.4.13.5.2. A sample with known LSD applied to it will be utilized as the control check to show the UV light box (tubes) is functioning properly. A control check will be performed monthly. Any discrepancies shall be noted and addressed (maintenance or fresh sample).

7.4.14.GHB

GHB (gamma-hydroxybutyrate) is a controlled substance in Idaho, while its precursors GBL (gamma-butyrolactone) and 1,4-Butanediol (BD) are not. This is problematic in that the interconversion of GBL to GHB and BD to GHB is simply pH dependent. In aqueous solutions, GHB and GBL will exist in equilibrium. The relative concentrations of each are also pH dependent.

- 7.4.14.1. Supplies (equipment/reagents/standards)
 - 7.4.14.1.1. GCMS and appropriate analytical software.

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- 7.4.14.1.2. pH paper.
- 7.4.14.1.3. Chloroform, methanol, ethanol, ethyl acetate or other appropriate solvents.
- 7.4.14.1.4. H_2SO_4 , Bis(trimethylsilyl)trifluoroacetamide with trimethylchlorosilane (BSTFA with 1% TMCS,a trimethylsilyl (TMS) derivatizing reagent).
- 7.4.14.1.5. Bromocresol green, methyl orange, dextrose, aniline hydrochloride, sodium hydroxide.
- 7.4.14.2. Method
 - 7.4.14.2.1. Color Test
 - 7.4.14.2.1.1. Bromocresol green mix 0.03g in 100 mL of 4:1 methanol:water. Adjust pH to 7 with NaOH.
 - 7.4.14.2.1.2. Methyl Orange mix 0.01g of Methyl orange in 100 mLof methanol. Adjust pH to 7 with NaOH.
 - 7.4.14.2.1.3. Modified Schweppes
 - Solution A mix 2.0g dextrose in 20 mL of water
 - Solution B mix 2.4g aniline hydrochloride in 20mLof ethanol.
 - Mix Solution A & B and dilute to 80 mL with methanol.
 - 7.4.14.2.1.4. Mix Bromocresol green solution with the Methyl orange solution in a 1:1 ratio. Add 3 parts of this combined solution to 1 part of the Schweppes reagent.
 - 7.4.14.2.2. Physical Tests
 - 7.4.14.2.2.1. Pure GBL and BD are viscous liquids at room temperature. BD will solidify when placed in a refrigerator (approximately 4°C) while GBL willnot.
 - 7.4.14.2.2.2. GBL is soluble in chloroform, BD is not.
 - 7.4.14.2.3. GCMS
 - 7.4.14.2.3.1. GHB cannot be analyzed directly on a GCMS as it will convert to GBL in the heated injection port. Add concentrated sulfuric acid to an aqueous sample, extract with chloroform and analyze. If GBL is detected, the sample must be derivatized with BSTFA before injection.

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7.4.14.2.3.2. GHB

- Extract aqueous sample with chloroform, discard chloroform.
- Dry down aqueous layer with nitrogen or dry air. Sample can be warmed to expedite drying as long as the temperature remains below 60° C.
- Once sample is completely dry, add 100-200 μL of BSTFA.
- Cap sample and heat at approximately 60-70° C for 15-20 minutes.
- Add ethyl acetate and analyze.

7.4.14.2.3.3. GBL

• Extract with chloroform and analyze.

7.4.14.2.3.4. BD

- If pure BD is suspected, dilute with methanol and inject.
- In aqueous samples, if concentrations of BD are high enough, extract with chloroform.
- Dry down sample, add methanol and analyze.
- BD will derivatize with BSTFA (add 100-200 μ L ofBSTFA, cap sample and heat at approximately 60-70° C for 15-20 minutes, add ethyl acetate and analyze).

7.4.14.3. Extraction scheme

7.4.14.3.1. Solids

- 7.4.14.3.1.1. Perform color test.
- 7.4.14.3.1.2. If negative, dissolve in methanol and analyze on GCMS.
- 7.4.14.3.1.3. If positive, follow 7.4.14.2.3.1.
- 7.4.14.3.1.4. If GCMS is negative, analysis is complete.
- 7.4.14.3.1.5. If GCMS has GBL, derivatize original sample with BSTFA and analyze on GCMS or run sample on FTIR.

7.4.14.3.2. Clear, thick liquids

- 7.4.14.3.2.1. Place 1-5 mL of sample in the freezer for 15 minutes. If it solidifies, extract with methanol and analyze on the GCMS. If results indicate the presence of GBL, proceed to section 7.4.14.3.3.4.
- 7.4.14.3.2.2. If sample remains a liquid, go to section 7.4.14.3.3.

7.4.14.3.3. Aqueous samples

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- 7.4.14.3.3.1. Perform color test.
- 7.4.14.3.3.2. Acidify a portion of the sample with concentrated H_2SO_4 and extract with chloroform. Analyze the chloroform layer on the GCMS. If results are negative for GBL, proceed with section 7.4.14.3.3.3. If GBL is present, skip to section 7.4.14.3.3.4. Report BD, if found.
- 7.4.14.3.3.3. If results are negative, take a portion of the original item and dry down with nitrogen or air and heat (below 60° C). Extract with methanol and analyze with GCMS.
- 7.4.14.3.3.4. Take a portion of the original item and extract with chloroform. Analyze the chloroform layer on the GCMS. Report GBL, if found.
- 7.4.14.3.3.5. If not GBL, analyze using 7.4.14.2.3.2. Report GHB, if found.
- 7.4.14.4. Interpretations
 - 7.4.14.4.1. A positive color reaction for the presence of GHB will turn green.
- 7.4.14.5. Cautions
 - 7.4.14.5.1. Aniline is acutely toxic, handle with care.
- 7.4.14.6. Controls
 - 7.4.14.6.1. Positive and negative controls shall be run with the color test and documented in the case notes.
 - 7.4.14.6.1.1. An authenticated GHB standard shall be used as a positive control.
 - 7.4.14.6.1.2. A clean well shall be used as a negative control.
- 7.4.15.0ther Controlled Substances
 - 7.4.15.1. If a specific method is not spelled out for analysis, it is up to the discretion of the analyst to determine the appropriate extraction to confirm the identity of the substance. This includes, but is not limited to, the analysis of khat, steroids, synthetic cannabinoids, substituted cathinones, fentanyl, or any number of designer drugs encountered in the laboratory.
- **7.5. Technical records** See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual
- 7.6. Evaluation of measurement uncertainty

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- 7.6.1. For balances used in weighing evidence, the measurement uncertainty (variability) of the balance has been determined by an in-house empirical performance study of that balance that includes the sources of uncertainty listed in 7.6.1 of the Quality Assurance Manual. The measurement uncertainty (MU) is calculated annually and updated (if necessary). The current uncertainty values can be found in the electronic laboratory documents. The uncertainty values and windows are posted near the balances.
- 7.6.2. Case notes shall indicate the uncertainty value for the balance(s) used when the total weight of an item(s) falls within the window of uncertainty at regulatory limits.
- 7.6.3. Measurement Uncertainty Calculations
 - 7.6.3.1. The "d" value of a balance is the readability of the balance or the smallest division displayed on the balance readout. The "d" value also shows the precision obtainable for the balance (e.g., precision to the 10ths, 100ths, or 1000ths).
 - 7.6.3.2. Regardless of what scale is used, uncertainty values not already in increments of "d" shall be rounded up to the nearest "d" increment. For example: If the uncertainty of a scale with d-value equal to 0.005 kg is 0.013 kg, then the uncertainty shall be rounded up to the nearest increment of "d" resulting in an uncertainty of 0.015 kg.
 - 7.6.3.3. The measurement result shall include the measured quantity value (X), along with the associated expanded uncertainty (U), and this measurement shall be reported as X (\pm) U, where U is consistent with the units of X. (Example: 28.05 g +/-0.04 g).
 - 7.6.3.3.1. For a single measurement, the reported weight of an exhibit shall be the balance reading \pm the calculated uncertainty.

Example 1: (assume a d-value of 0.01)

The reading from the scale is 1.23g and the uncertainty value is 0.01. The reported value shall be $1.23g \pm 0.01g$.

- 7.6.3.3.2. For multiple measurements, one cannot simply add all of the uncertainties together and report the combined uncertainties as the total uncertainty. This is because the error is random; sometimes the measured weight will be higher and other times it will be lower than the true value.
- 7.6.3.3.3. To calculate the Uncertainty (U) for multiple measurements with the same uncertainty, Equation 1 must be applied.

Equation 1 $U = \sqrt{N x (u_b)^2}$

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U = total Uncertainty for the sum value

N = number of measurements

 u_b = uncertainty of the balance

Equation 1 can be simplified to Equation 2

Equation 2
$$U = \sqrt{N} \times u_b$$

Example 2:

A balance used to obtain the weight for five bindles has an uncertainty of 0.01 gram. Table 2 shows the weights of the 5 bindles using this balance.

Table 3

Bindle #	Weight (g)
1	0.52
2	0.67
3	0.78
4	1.02
5	0.21
Total	3.20 g

The uncertainty from each weighing shall be considered. To determine the combined uncertainty from all five weighings, use Equation 2.

Using Equation 2, the combined uncertainty would be calculated as follows:

$$U = \sqrt{N} \times u_b = \sqrt{5} \times 0.01 = 0.022g$$

Since uncertainty values will have the same precision as the balance being used and shall be rounded up to the nearest increment of "d" (d=0.01), the calculated total uncertainty value of 0.022 grams would be round up to 0.03 grams.

The final weight shall be reported out as $3.20 \text{ grams} \pm 0.03 \text{ grams}$.

Example 3: (significant figures and d-values)

A case has ten bags of marijuana, each weighed separately:

Total Weight: 52.015 kg Scale Uncertainty: 0.005 kg

d-value: 0.005 kg

Using Equation 2, the combined uncertainty would be:

$$U = \sqrt{N} \times u_b = \sqrt{10} \times 0.005 = 0.0158 \text{ kg}$$

This combined uncertainty value would be rounded up to the precision of the scale (i.e., 0.016 kg) and then up to the nearest increment of "d", making the final uncertainty 0.020 kg.

The final weight shall be reported as 52.015 kg ± 0.020 kg.

7.6.3.3.4. At times, individual items within an exhibit will necessitate using two different balances or different weights may fall within different calculated uncertainty values for a particular balance (see note below). The example below would apply to either of these situations. These calculations also apply to subtraction calculations; although the difference is found through subtraction, the combined uncertainty is the sum of the separate uncertainties. To calculate the Uncertainty (U) for multiple measurements with more than one U value, see example below.

Example 4:

A submission came into the laboratory that consisted of four plastic bags and three paper bags all containing material that was visually consistent. Due to the different sizes of the containers, the analyst decided to weigh the contents of the plastic bags on a 400-gram capacity balance with an uncertainty value of 0.01 grams and weigh the contents of the paper bags on a larger capacity balance with an uncertainty value of 0.4 grams.

As an aside, if the analyst made all the measurements on a single 600-gram capacity scale that had separate uncertainty values for different ranges of weights, the "d" value would be the same for both weight levels.

Table 4: Balance B1

Uncertainty = 0.01 g, "d" = 0.01

Plastic bags	Weight (g)
1	10.56
2	14.66

3	28.50
4	31.27
Total	84.99

Table 5: Balance B2

Uncertainty = 0.4 g, "d" = 0.2

Paper bags	Weight (g)
1	450.6
2	422.8
3	575.2
Total	1448.6 g
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	l .

For this calculation Equation 2 shall be used twice – once for B1 and once for B2.

The combined equation will be as follows:

Equation 3
$$U_{tot} = (\sqrt{N_{B1}} \times u_{B1}) + (\sqrt{N_{B2}} \times u_{B2})$$

 U_{tot} = total uncertainty

 N_{B1} = number of measurements on Balance 1

 u_{B1} = uncertainty of Balance 1

 N_{B2} = number of measurements on Balance 2

 u_{B2} = uncertainty of Balance 2

Using the above numbers for the weights obtained in Table 4 and Table 5, the following measurement of uncertainty is obtained.

$$U_{\text{tot}} = (\sqrt{4} \times 0.01) + (\sqrt{3} \times 0.4) = 0.71 \text{ grams}$$

The combined net weight (total of balance B1 plus total of balance B2) is 1533.59 grams. When combining measurements with different degrees of precision, the precision of the final answer can be no greater than the least precise measurement. With this rule in mind, the total weight would be 1533.5 grams (one decimal place). The weight itself is never rounded up, but rather is truncated. The calculated uncertainty (U) equaled 0.71 gram, which

would be rounded up to 0.8 gram (uncertainty sum rounded up to the nearest increment of the larger "d" value).

The reported net weight shall be 1533.5 grams ± 0.8 grams.

7.6.4. The reported coverage probability of the expanded uncertainty is approximately 95%.

7.7. **Ensuring the validity of results**

- 7.7.1. A positive and negative control shall be run to verify the reagent preparation. This control shall occur before use or, if appropriate, concurrent with the test.
 - The positive control shall be performed by testing the reagent with a substance that will react with the reagent to produce a known color.
 - 7.7.1.2. A negative control shall be performed by testing the reagent with a substance known not to react or with a blank spot well.
 - The results of these tests shall be recorded, along with the date the reagent 7.7.1.3. was made, the initials of the preparer, and if the reagent worked as expected. This shall be recorded on the reagent log.
 - 7.7.1.4. All color reagents that are made in bulk shall be checked monthly and the results of the checks shall be recorded in the reagent log.
 - Bulk reagents include: Marquis, Simons (Secondary Amine), Cobalt 7.7.1.4.1. Thiocyanate and Duquenois-Levine.
 - 7.7.1.4.2. A methamphetamine standard shall be used for the positive control for the Marquis and Simons color reagent. A cocaine standard shall be used for the positive control for the Cobalt Thiocyanate color reagent. A marijuana standard shall be used for the positive control for the Duquenois-Levine reagent.
 - Dimethyl sulfone shall be used for all the negative controls except 7.7.1.4.3. for the Duquenois-Levine reagent. A blank test tube will be used for its negative control.
 - The control results for reagents that are prepared for one-time use (or not made in bulk) shall be documented in the case notes.
 - 7.7.1.6. In the case notes, a "pass" indicates both the controls for the standard and the blank worked properly. A "fail" indicates one or both controls were unsuccessful.
 - Reagents prepared in the laboratory shall be labeled with the identity of the reagent, date of preparation and/or lot number, and the identity of who made the reagent.

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- 7.7.1.7.1. For a prepared reagent, the lot number shall be the date and the initials of the analyst who prepared it (i.e. 070716HC).
- 7.7.1.8. The components used in the preparation of the reagent shall be documented on the reagent log, as well as who made the reagent.
- 7.7.1.9. If at any time a reagent does not work as expected, re-make the reagent and perform a control check. If it still fails, the components used in the preparation and the controls will be evaluated and/or replaced until the reagent passes.
- 7.7.1.10. There is no expiration date for reagents. They can be used as long as the controls react appropriately.
- 7.7.2. In drug chemistry, the reference materials are the drug standards (primary or secondary) that are used as a reference for confirmatory analysis, color tests, TLC and the UV light box.
 - 7.7.2.1. Primary standards shall be obtained from an accredited supplier, if available.
 - 7.7.2.2. Secondary standards are laboratory produced samples or case work items whose composition have been verified by comparison of its Category A instrumental data (e.g., GC/MS, FTIR) to that of a primary standard or a scientifically accepted literature reference.
- 7.7.3. Before a drug standard can be used as a reference for casework, it shall be authenticated, either on the GCMS or FTIR. This will confirm competence or traceability for the supplier and product.
 - 7.7.3.1. A standard shall be considered authenticated when the library match is greater than 85%. If the match is less than 85%, then two chemists must concur on the validity of the match and initial the printout. This printout shall be kept in the standards logbook or saved electronically.
 - 7.7.3.2. Reference libraries can come from any reliable source, including, but not limited to, instrument libraries, scientific journals or publications. When a reference is not available, mass spectral interpretation may be used in conjunction with the Certificate of Analysis (COA). This would apply in cases where instrumental data for a drug metabolite or isomer is not yet published, but a structurally similar compound is available to assist with interpretation. Again, this would require two chemists to concur on the interpretation and initial the printout and the printout shall be kept in the standards logbook or saved electronically.
- 7.7.4. Primary standards shall be uniquely identified by the manufacturer and lot number. For secondary standards, the case number (and exhibit number, if applicable) shall be used as the lot number.
- 7.7.5. All drug standards shall be inventoried. A Drug Standard Form shall be filled out for all standards that have more than a one-time use.

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- 7.7.5.1. The form shall list the drug, schedule, source, initial gross weight, current weight and date of authentication.
- 7.7.6. Annually, the standards shall be inventoried. This shall be documented on the Drug Standard Form.
- 7.7.7. Drug standards shall be securely stored in the laboratory in a double locking key box. Access to the standards shall be limited to personnel designated by the laboratory manager. Two designated personnel are required to open this box at any given time. If it is not possible to secure the drug standards in a double lock box, they will be maintained in a sealed state and their access will be tracked (i.e. opioid or fentanyl kits).
- 7.7.8. A logbook shall be maintained for the standards double locking key box that lists the standard and the date and initials of personnel accessing it. Personnel accessing drug standards that are not in a double lock box need to document this on the double lock box logbook.
- 7.7.9. Documentation for the standards shall also include the DEA Form-222 and receipt of purchase.

7.8. Reporting of results

- 7.8.1. In addition to the reporting requirements outlined in the Quality Assurance Manual, a controlled substance report shall also contain the original weight or number of plants, pills/capsules, and the schedule of the drug, if applicable (weight/volume of liquids is not reported).
- 7.8.2. All items received, including items which were not analyzed, shall be noted in the report.
- 7.8.3. For any weight that registers 0.09 grams or less on the balance, it shall be reported as "< 0.10 grams".
- 7.8.4. If the item is not weighed (and can't be counted), due to a small amount, the report shall have "residue" in the description of the item.
- 7.8.5. If an extract(s) must be returned with the evidence (per 7.4.6.2 and 7.4.6.3) the report shall state "**Remaining extract(s) was/were returned with evidence**."
- 7.8.6. If measurement uncertainty is reported, the measurement result shall include the measured quantity value (X) along with the associated expanded uncertainty (U) and shall be reported as X +/- U, where U is consistent with the units of X. (Example: 28.05 g +/- 0.04 g).
- 7.8.7. If measurement uncertainty is reported, the report shall also state "**The uncertainty was calculated at approximately the 95% confidence level**".
- 7.8.8. If a substance cannot be identified after meeting the minimum examination

requirement then "No controlled substance(s) detected" shall be reported.

- 7.8.9. If a controlled substance is present but not confirmed, the report shall read "Preliminary results indicate the presence of______, not confirmed".
 - 7.8.9.1. The compound and/or class of compounds shall be reported.
 - 7.8.9.2. The reason why the substance was not confirmed shall be on the report.
 - 7.8.9.2.1. **Exception:** When an item is brought into the lab to be tested for a preliminary result only, a "Preliminary Results" report shall be issued. It is not necessary to include a reason why it was not confirmed.
- 7.8.10.Non-analytical identifications of pills shall read, "**Source lists as**______". If the relative amounts of the therapeutic ingredients will affect the scheduling, then they shall be recorded on the report.
- 7.8.11.If there is limited sample to test during the course of the examination and/or the analyst is unable to satisfy the minimum examination requirement then "**Insufficient sample for identification**" shall be reported.
- 7.8.12. For synthetic cannabinoids that have ambiguous scheduling, the report shall read, "______, a synthetic cannabinoid."
- 7.8.13.If sampling occurs, follow the relevant reporting guidelines in the Quality Assurance Manual.
- **7.9. Complaints** See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual
- **7.10. Nonconforming work** See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual
- 7.11. Control of data and information management
- 8. Management system requirements
 - 8.1.Options See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual
 - **8.2.Management system documentation (Option A)** See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual
 - **8.3.Control of management system documents (Option A)** See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual
 - 8.4. Control of records (Option A)
 - 8.4.1. The documentation needed to support the conclusion(s) in the report shall be kept in the case file. Current batch documentation shall be stored in an area of the laboratory known to and accessible to the drug chemists.

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- 8.4.2. If an observation, data or test result is rejected, the person rejecting it shall state the reason why it was rejected and initial and date the reason.
 - 8.4.2.1. This documentation (i.e. chromatograph) will be retained in the case file or it will be documented in the notes. If the original data is not retained in the case file, it will be archived.
- 8.4.3. Authentication documentation shall be kept for each standard.
- 8.4.4. GCMS and FTIR instrumental data files shall be backed up monthly.
- 8.4.5. A maintenance log shall be kept for equipment. Entries into the log should include any symptoms and type of maintenance performed.
- 8.4.6. The approved abbreviation list for the drug chemistry discipline can be found in the electronic lab documents. Commonly accepted abbreviations or abbreviations that are readily recognizable to a reviewer do not need to be listed, such as those for metrics or elements. Chemical formulas do not need to be listed as abbreviations, but shorthand notations such as MeOH, for methanol, do. Care should be taken when isomers may be involved. For the designer drugs, a chemist should refer to professional literature for the accepted abbreviation.
 - 8.4.6.1. Should an analyst use an abbreviation that is not listed in the abbreviation list, then a reference shall be included in the case notes as to what the abbreviation means.
- 8.4.7. Records shall be stored and retained in such a way that they are readily retrievable.
- **8.5. Actions to address risks and opportunities (Option A)** See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual
- **8.6.Improvement (Option A)** See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual
- **8.7.Corrective actions and quality incidents (Option A)** See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual
- **8.8.Internal audits (Option A)** See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual
- **8.9.Management reviews (Option A)** See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual

Ada County Sheriff's Office Forensic Lab History of Drug Chemistry Analytical Method

SECTION AND COMMENTS	DATE ADOPTED	AUTHOR	REVIEWER(S)
Original document	7/29/19	H.Campbell	
Manual has been revised to fit the new numbering system 17025:2017 3.4.5/3.4.6 added palmate and pinnate to terms and definitions 3.5.9 Clarified definition of sample 6.3.6 added warning about emptying helium gas tank	12/8/20	H Campbell	N. Wheatley S. Guess K. Brown
6.3.7 added, Methods that reference temperatures are all approximate. Temperatures do not have to be recorded or monitored.6.4.1.1.5 deleted alternate light source6.4.5.6 added statement to review and address cases that may be			
affected if a balance is taken out of service 6.4.6.6.4.4 added approximately in front of 10% 6.4.7 FTIR, removed sections that didn't apply to an ATR, updated			
controls 6.4.7.2.2 removed authenticated since may be authenticating standard on instrument 6.6.1.4 added "Reference materials shall be purchased from an			
accredited supplier, if available". 7.3.1.3 added the sampling plan used does not need to be in report 7.3.1.3.5 added non-relevant items that are described in QAM			
7.3.1.3.6 changed "notes" to "report" 7.4.1.1.2 added info about documentation images 7.4.4.1 added Category B and/or C are preliminary/presumptive tests			
7.4.4.2 added "from Category B and/or C" 7.4.4.7 changed "sample" to "sample extract" 7.4.4.9 added FTIR will be compared to internal library 7.4.4.10.3 added for clarification			
7.4.4.12 changed findings and conclusions to opinions and interpretations 7.4.9.2.1 removed the section that the monthly control checks will be			
recorded in the reagent log and added it to 7.7.1.4. 7.4.9.5.2 added "Most color tests work relatively quickly, depending on			
the concentration of the sample. Be aware that many color reagents are made with a strong acid that will eventually char/burn the sample and appear brown/black in color".			
7.4.11.2.3/7.4.11.2.5.1 added "residue/sample" to try to include all types of marijuana samples-originally only mentioned plant material 7.4.11.3.2 added unicellular hairs			
7.4.11.4.1 removed Duq-Lev test since now testing monthly 7.4.11.3.5.4 added "positive" 7.4.12.3.2 added "unless differentiated by TLC, then the compound			
identified will be reported". 7.4.13.2.1.3 Added pDMAB color test for LSD preliminary test			

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7.4.13.2.1.4 Added TLC as a preliminary test for LSD and added		
approximately in front of temperature		
7.4.13.4.2 added caution about light and heat degrading LSD		
7.4.13.5.2 added a control check for the UV light box, perform monthly		
7.4.14.2.2.1 added approximately in front of temperature		
7.4.14.2.3.2 added approximately in front of temperature		
7.4.14.3.1.3 Changed to 7.4.14.2.3.1 to make sense		
7.4.14.2.3.4 added approximately in front of temperature		
7.6.1 added "that includes the sources of uncertainty listed in 7.6.1 of		
the Quality Assurance Manual"		
7.7.1.4.1 included Duq-Lev in the bulk to be tested monthly, not with		
each use		
7.7.1.10 added no expiration date for reagents		
7.7.2 added drug standards used as a reference for color tests, TLC and		
UV light box		
7.7.2.2 removed Raman		
7.7.7 changed "all" to "drug" and added that drug standards that can't		
be put into double lock box will be sealed and access tracked		
7.7.8 added "standard" and if accessing sealed standards that this		
needs documented on the logbook for the double lock box		
7.8.13 added to follow QAM for reporting if sampling and added the		
word "relevant"		
8.4.2.1 changed wording to original data will be archived instead of		
chromatograph		
8.4.5 changed gcms/ftir to equipment		
0.4.5 changed genis/thr to equipment		

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